U.S. DEPARTMENT OF COMMERCE

PB80-161706

Stack Sampling Technical Information
A Collection of Monographs and Papers
Volume IV

(U.S.) Environmental Protection Agency
Research Triangle Park, NC

Oct 78

U.S. Department of Commerce
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TECHNICAL REPORT DATA (Please read Instructions on the reverse before completing)		
1. REPORT NO. EPA-450/2-78-042d	2.	3. RECIPIENT'S ACCESSION NO. PB80 161706
4. TITLE AND SUBTITLE Stack Sampling Technical Information: A Collection of Monographs and Papers Volume IV		5. REPORT DATE October 1978
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) Roger T. Shigehara (Editor)		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Environmental Protection Agency Emission Standards and Engineering Division Emission Measurement Branch Research Triangle Park, NC 27711		10. PROGRAM ELEMENT NO.
		11. CONTRACT/GRANT NO.
12. SPONSORING AGENCY NAME AND ADDRESS Same as above.		13. TYPE OF REPORT AND PERIOD COVERED
		14. SPONSORING AGENCY CODE
15. SUPPLEMENTARY NOTES		

16. ABSTRACT

"Stack Sampling Technical Information" is a four-volume collection of monographs and papers which have been compiled by the Emission Measurement Branch, ESED, OAQPS. The information specifically relate to current EPA test methods and compliance test procedures. The data presented in some of these documents have served as the basis for a number of revisions made in the EPA Reference Methods 1 through 8. Several of the documents are also useful in determining acceptable alternative procedures.

17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Gas Sampling Filtered Particle Sampling Gas Analysis	Stack Sampling	14B 14D
18. DISTRIBUTION STATEMENT Unlimited	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES
	20. SECURITY CLASS (This page) Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

United States
Environmental Protection
Agency

Office of Air Quality
Planning and Standards
Research Triangle Park NC 27711

EPA-450/2-78-042d
October 1978

PB80-161706

Air

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Stack Sampling Technical Information
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SPRINGFIELD, VA. 22161

EPA-450/2-78-042d

Stack Sampling Technical Information
A Collection of Monographs and Papers
Volume IV

Emission Standards and Engineering Division

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Air, Noise, and Radiation
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

October 1978

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Publication No. EPA-450/2-78-042d

PREFACE

The Clean Air Act of 1970 requires the Administrator of the Environmental Protection Agency to establish national emission standards for new stationary sources (Section 111) and hazardous air pollutants (Section 112). The development of these emission standards required the concurrent development of reference test methods and procedures. The reference test methods and procedures are published in the Federal Register along with the appropriate regulations.

From time to time, questions would surface concerning the methods and procedures. In many cases, specific studies would be needed to provide informed, objective answers. The papers and monographs resulting from these studies were usually distributed to people involved in emission measurement; a major method of distribution has been the Source Evaluation Society Newsletter.

To provide a readily available resource for new and experienced personnel, and to further promote standardized reference methods and procedures, it has been decided to publish the papers and monographs in a single compendium. The compendium consists of four volumes. The Table of Contents for all four volumes is reproduced in each volume for ease of reference.

Congratulations and sincere appreciation to the people who did the work and took the time to prepare the papers and monographs. For the most part the work was done because of personal commitments to the development of objective, standardized methodology, and a firm belief that attention to the details of stack sampling makes for good data. The foresight of Mr. Robert L. Ajax, the former Chief of the Emission Measurement Branch and now the Assistant Director, Emission Standards and Engineering Division, in providing the atmosphere and encouragement to perform the studies is gratefully acknowledged. The skill and dedication of Mr. Roger Shigehara, in providing personal supervision for most of the work, is commended.

Don R. Goodwin
Director
Emission Standards and
Engineering Division

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RECOMMENDED PROCEDURE FOR SAMPLE TRAVERSES IN DUCTS SMALLER THAN 12 INCHES IN DIAMETER

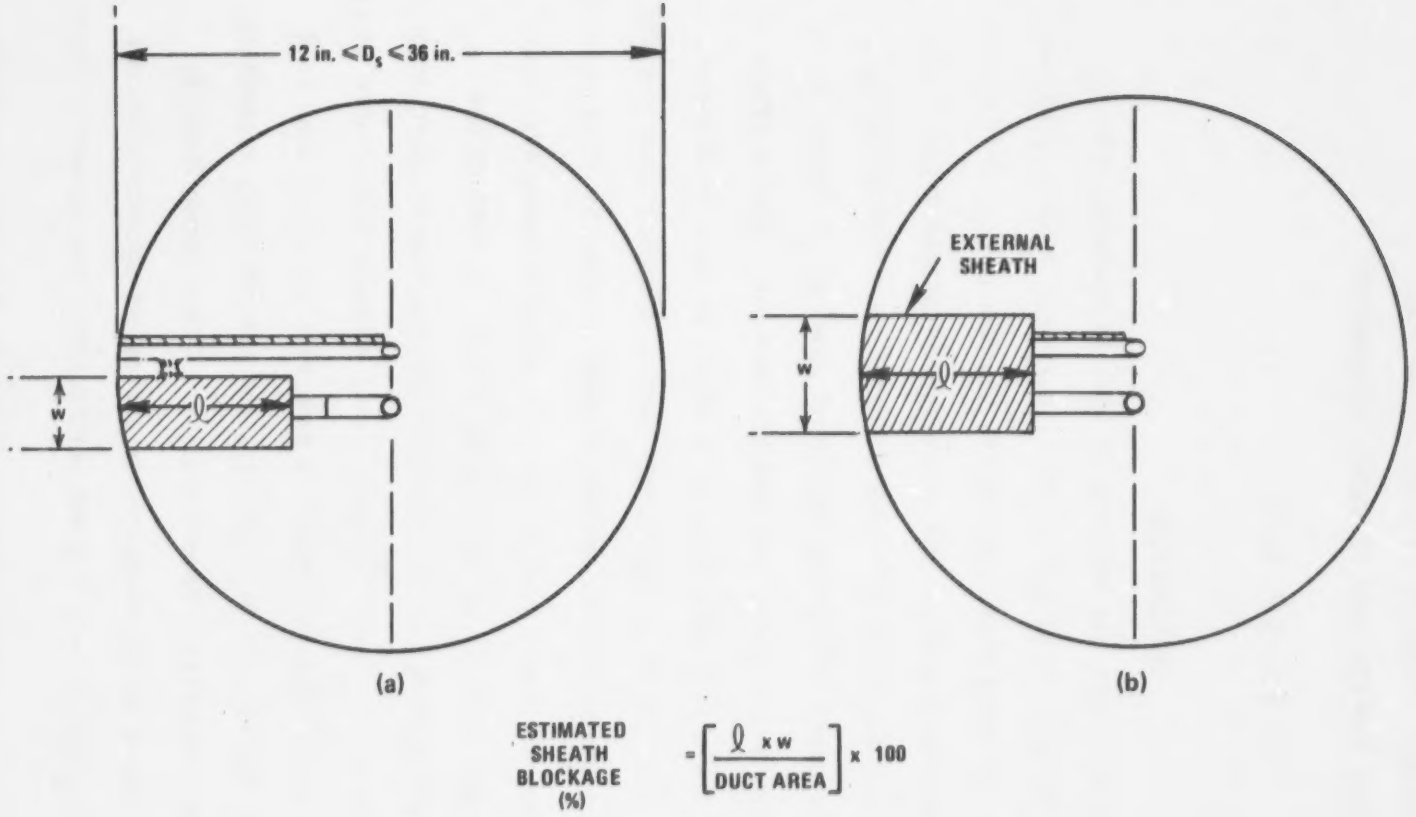
Robert F. Vollaro**

INTRODUCTION

In source sampling, stack gas velocity is usually measured with a Type-S pitot tube. In many field applications, the pitot tube is attached to a sampling probe, equipped with a nozzle and thermocouple. This combination is called a pitobe assembly. Most conventional pitobe assemblies* have a cylindrical sampling probe of 1-inch diameter, but, occasionally, an assembly has an external cylindrical sheath of about 2-1/2 inches in diameter, encasing the probe, pitot tube and thermocouple. When a pitobe assembly is used to traverse a duct that is 36 inches or less in diameter, the pitobe assembly can "block" a significant part of the duct cross section, as illustrated in the projected-area models, Figures 1a and 1b. This reduction in the effective cross-sectional area of the duct causes a temporary, local increase in the average velocity of the flowing fluid. In most pitobe assemblies, the impact opening of the Type-S pitot tube liesin approximately the same plane as the probe sheath (Figure 2) and, whenever appreciable sheath blockage exists, velocity head (ΔP) readings made with the pitot tube tend to reflect the local increase in gas velocity, and are not truly representative of the mainstream velocity. Recent studies[1, 2] have shown that, for sample traverses in ducts having diameters or equivalent diameters between 12 and 36 inches, blockage effects are not particularly severe, and a simple

*Designed according to the specifications outlined in APTD-0581 (Reference 3), or allowable modifications thereof.

** Emission Measurement Branch, ESED, OAQPS, EPA, RTP, NC, January 1977



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{l \times w}{\text{DUCT AREA}}\right] \times 100$$

Figure 1. Projected-area models for typical pitobe assemblies; shaded area represents approximate average sheath blockage for a sample traverse.

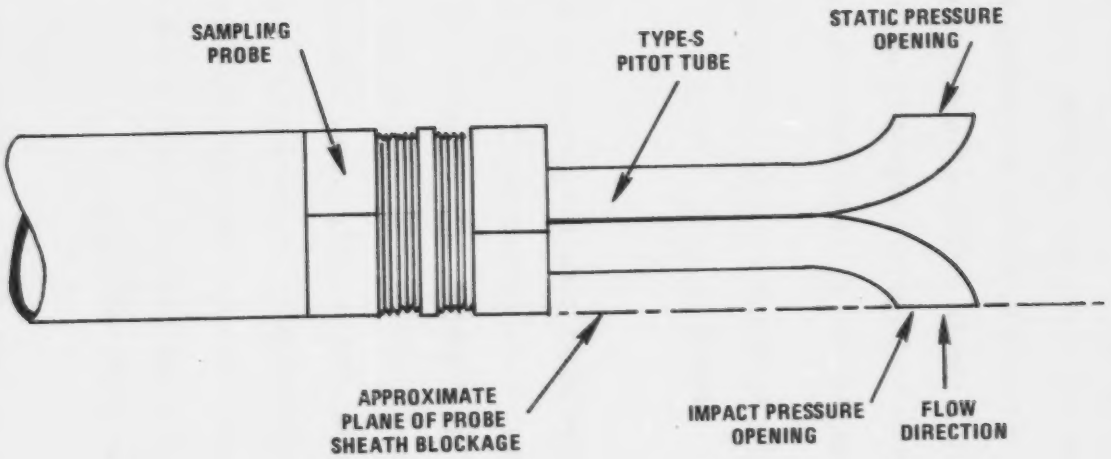


Figure 2. Type-S pitot tube, attached to a sampling probe, showing that the pitot impact opening and probe sheath lie in approximately the same plane.

adjustment in the value of the Type-S pitot tube coefficient (C_p) can be made to compensate for the pseudo-high ΔP readings (Figure 3). When the duct diameter (D_s) is less than 12 inches, however, probe sheath blockage effects intensify, and the adjustment technique illustrated in Figure 3 no longer applies. Therefore, alternative methodology must be used in order to obtain representative sample traverses in ducts of this size. The purpose of this paper is to propose a method by which satisfactory sample traverses can be conducted when D_s is between 4 and 12 inches.

PROPOSED METHOD FOR SAMPLE TRAVERSES WHEN 4 in. $\leq D_s <$ 12 in.

METHODOLOGY

To conduct representative sample traverses in ducts having diameters between 4 and 12 inches, it is recommended that the arrangement illustrated in Figure 4 be used. In Figure 4, velocity head (ΔP) readings are taken downstream of the actual sampling site. The purpose of the straight run of duct between the sampling and velocity measurement sites is to allow the flow profile, temporarily disturbed by the presence of the sample probe, to redevelop and stabilize. The pitot tube and sampling nozzle shown in Figure 4 are different from those of a conventional pitobe assembly;[3] construction details of these components are discussed below.

A. Pitot tube.

A standard pitot tube shall be used, instead of a Type-S, to monitor stack gas velocity. When D_s is less than 12 inches, a Type-S pitot tube can begin to block a significant part of the duct cross section and yield

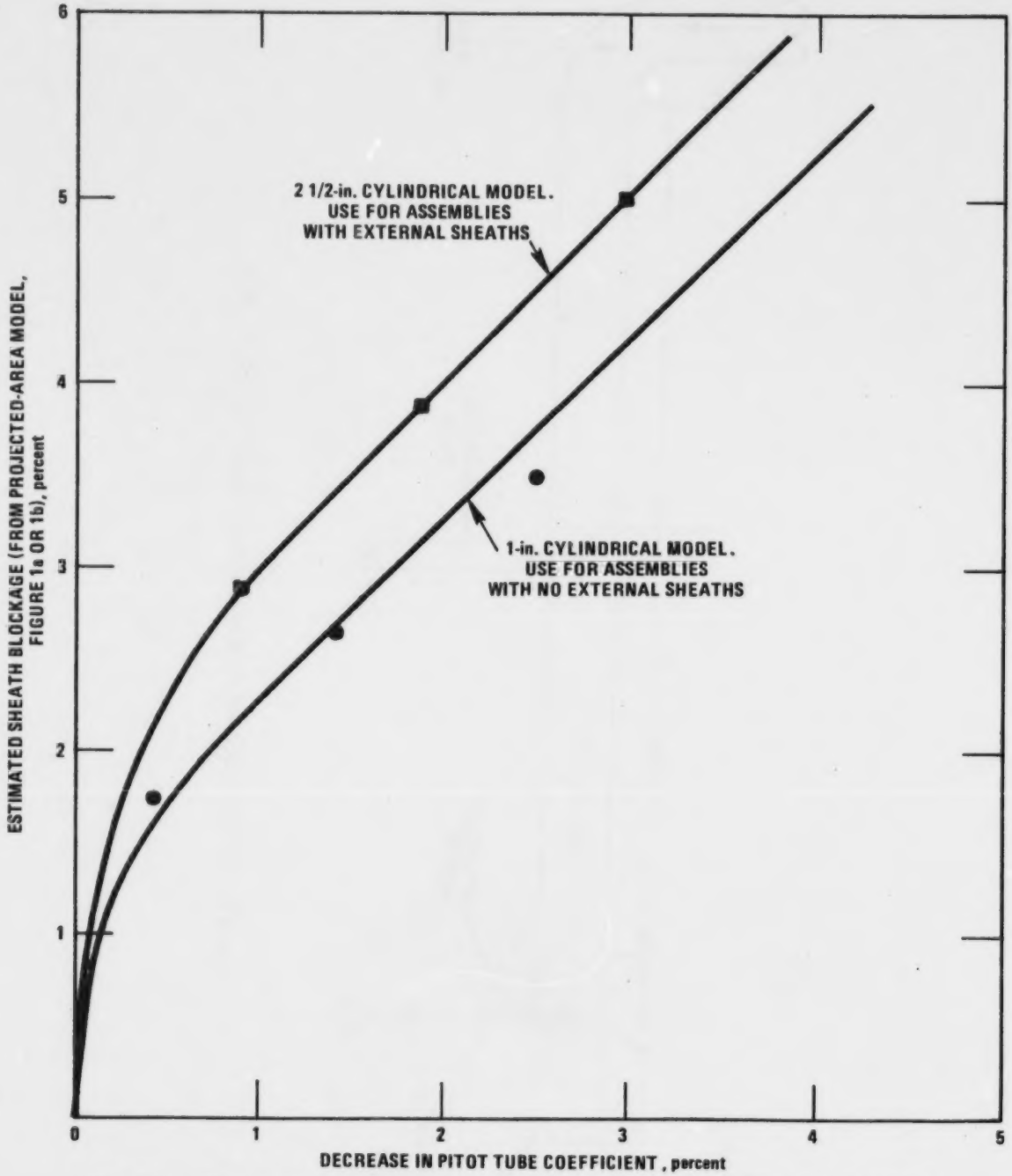


Figure 3. Adjustment of Type-S pitot tube coefficients to account for sheath blockage ($12 \text{ in.} \leq D_S \leq 36 \text{ in.}$).

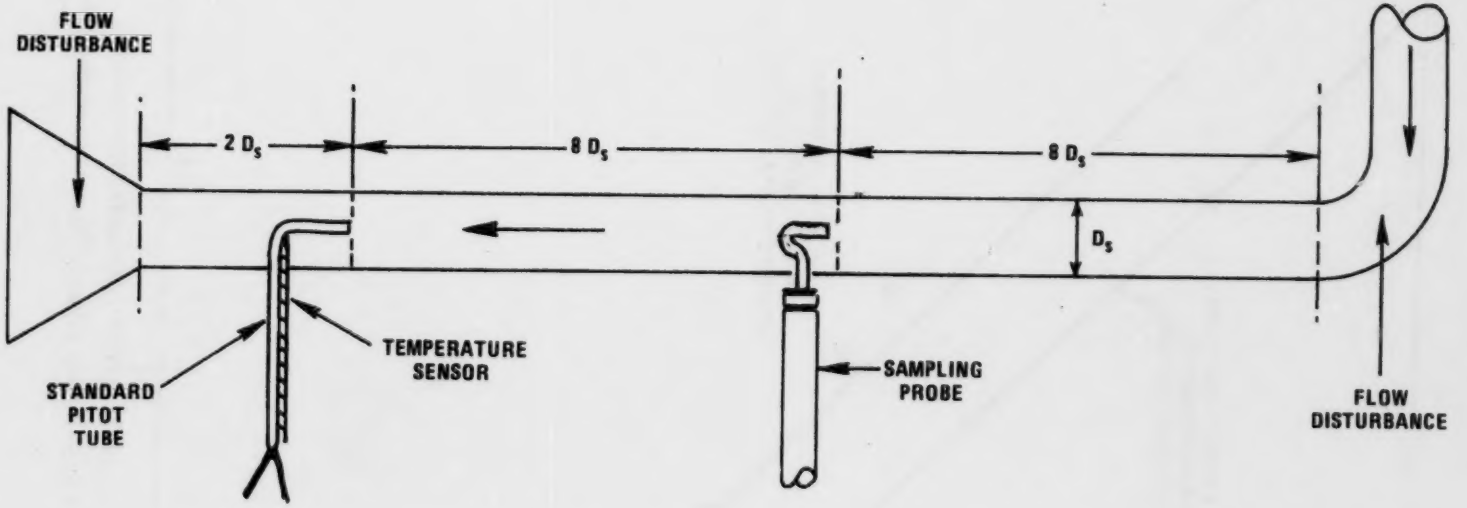


Figure 4. Recommended sampling arrangement, when 4 in. $\leq D_s < 12$ in.

pseudo-high ΔP values. Cross-section blockage is not a serious problem with a standard pitot tube, however, for two reasons: (1) the impact and static pressure openings of a standard pitot tube, unlike those of a Type-S, follow a 90° bend, and are located well upstream of the stem of the tube (compare Figures 2 and 5); and (2) when properly aligned, the sensing head of a standard pitot tube is parallel, not perpendicular, to the flow streamlines in the duct.

The preferred design for the standard pitot tube is the hemispherical-nosed design (Figure 5). Pitot tubes constructed according to the criteria illustrated in Figure 5 will have coefficients of 0.99 ± 0.01[4, 5]. Note, however, that for convenient tubing diameters (dimension "D" Figure 5), the static and impact sensing holes of the hemispherical-type pitot tube will be very small, thus making the tube susceptible to plugging, in particulate or liquid droplet-laden gas streams. Therefore, whenever these conditions are encountered, either of the following can be done: (1) a "back purge" system of some kind can be used to clean out, periodically, the static and impact holes; or (2) a modified hemispherical-nosed pitot tube (Figure 6), which features a shortened stem and enlarged impact and static pressure holes, can be used instead of the conventional hemispherical type. It has recently been demonstrated that the coefficients of the conventional and modified hemispherical-nosed tubes are essentially the same.[6]

B. Sampling nozzle.

The sampling nozzle can either be of the buttonhook or elbow design. The nozzle shall meet the general design criteria specified in Section 2.1.1 of the revised version of EPA Method 5, except that the entry plane of the

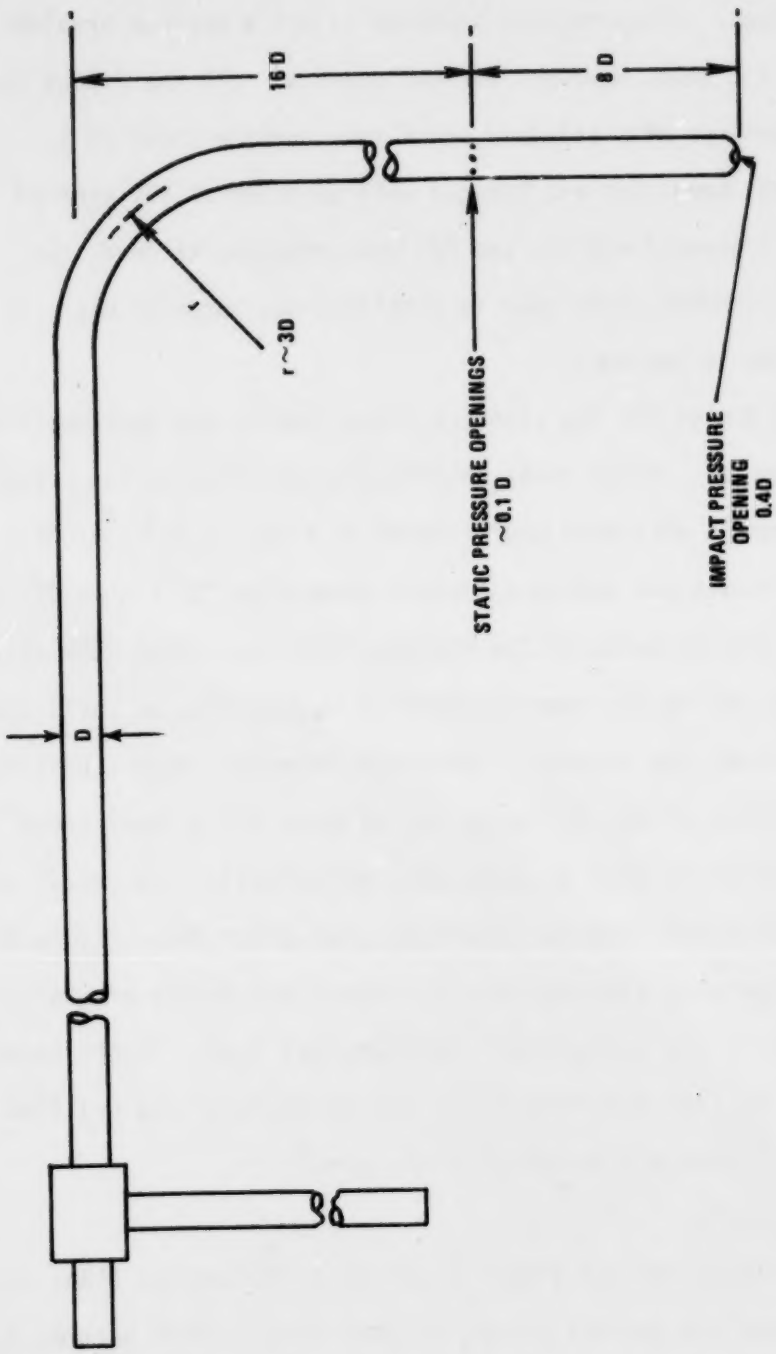


Figure 5. Hemispherical-nosed standard pitot tube.

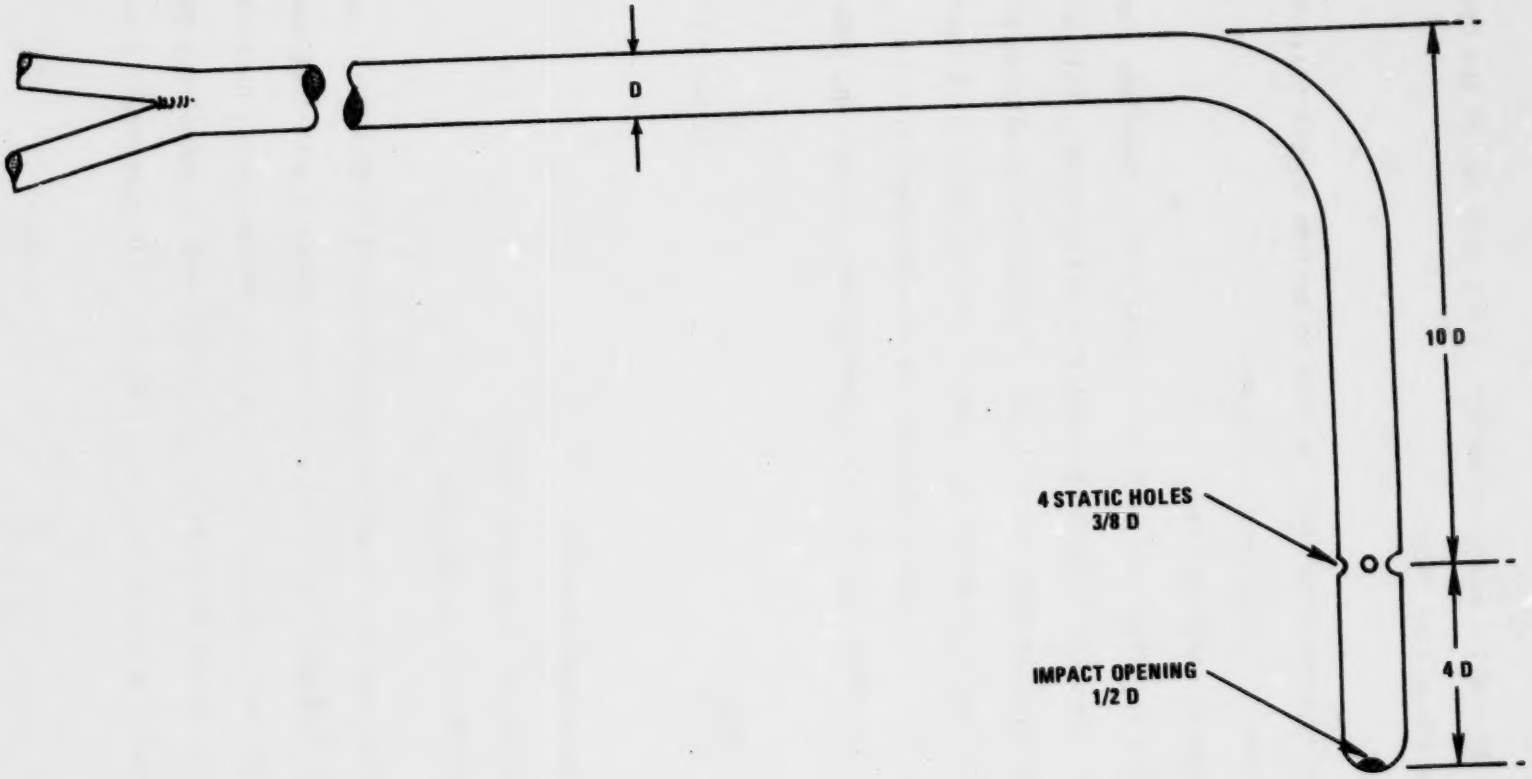


Figure 6. Modified hemispherical-nosed pitot tube.

nozzle must be at least 2 nozzle diameters (i.d.) upstream of the probe sheath blockage plane (see Figure 7).

PROCEDURES

The following procedures shall be used to perform sample traverses using the arrangement illustrated in Figure 4:

A. Location of sampling site.

Select a sampling site that is at least 8 duct diameters downstream and 10 diameters upstream from the nearest flow disturbances; this allows the velocity measurement site to be located 8 diameters downstream of the sampling location and 2 diameters upstream of the nearest flow disturbance (see Figure 4). For rectangular stacks, use an equivalent diameter, calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{L + W} \qquad \text{(Equation 1)}$$

Where:

D_e = Equivalent diameter

L = Length of cross section

W = Width of cross section

If a sampling site located 8 diameters downstream and 10 diameters upstream from the nearest disturbances is not available, select a site that meets these criteria as nearly as possible. Under no circumstances, however, shall a sampling site be chosen which is less than 2 diameters downstream and 2.5 diameters upstream from the nearest disturbances; this guarantees a minimum

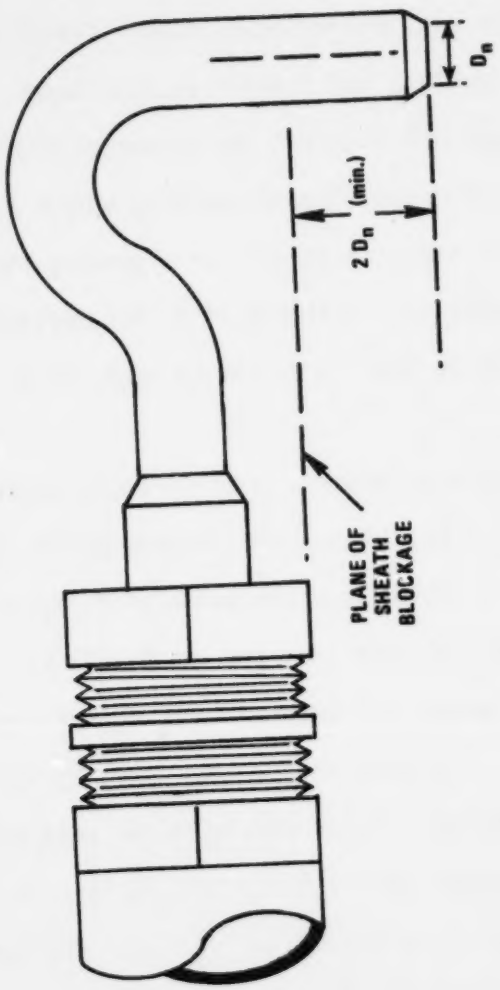


Figure 7. Recommended sampling nozzle design for use when 4 in. $\leqslant D_s < 12$ in.

of 2 diameters of straight run between the sampling and velocity measurement sites, and 0.5 diameters between the velocity measurement site and the nearest flow disturbance.

B. Number of traverse points.

The correct number of traverse points shall be determined from Figure 8. To use Figure 8, proceed as follows: first, determine the three distances, "A", "B", and "C", and express each distance in terms of duct diameters; second, read from Figure 8 the number of traverse points corresponding to each of these three distances; third, select the highest of the three numbers of traverse points, or a greater number, so that for circular ducts the number is a multiple of 4; for rectangular ducts, the number should be chosen so that it is one of those shown in Table 2.

C. Location of traverse points, circular cross sections.

For circular stacks, locate the traverse points on 2 perpendicular diameters, according to Table 1 and the example of Figure 9a. Any traverse point located less than 1/2 inch from the stack wall will not be acceptable for use as a sampling point; all such traverse points shall be "adjusted" by relocating them to a distance of 1/2 inch from the wall. In some cases, this relocation process may involve combining two adjacent traverse points to form a single "adjusted" point; thus, in some instances, the number of points actually used for sampling may be less than the number of traverse points obtained from Figure 8.

D. Location of traverse points, rectangular cross sections.

For rectangular stacks, divide the cross section into as many equal rectangular elemental areas as traverse points (as

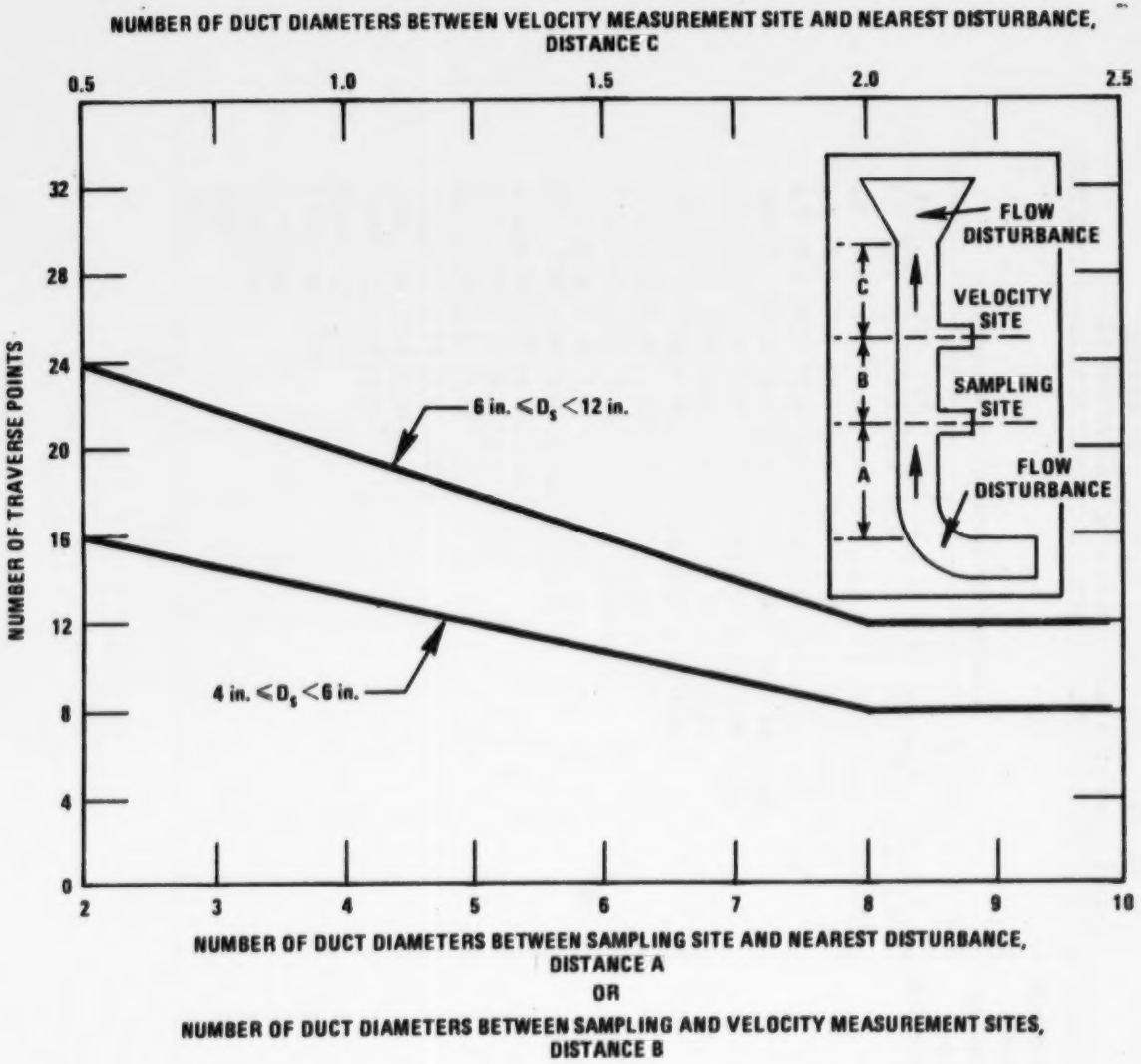


Figure 8. Minimum number of traverse points, 4 in. ≤ D_s < 12 in.

Table 1. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS (PERCENT OF STACK DIAMETER FROM INSIDE WALL TO TRAVERSE POINT)

Traverse point number on a diameter	Number of traverse points on a diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2
14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

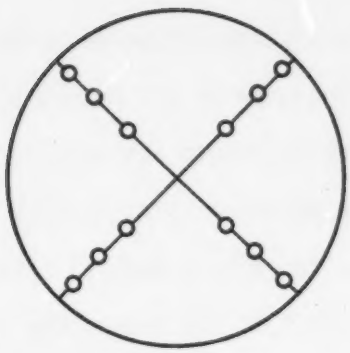


Figure 9a. Cross section of circular stack divided into 12 equal areas, showing location of traverse points.

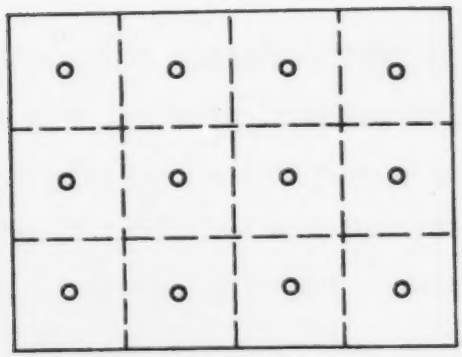


Figure 9b. Cross section of rectangular stack divided into 12 equal areas, with traverse points at centroid of each area.

determined in Section "B" above), according to Table 2. Locate a traverse point at the centroid of each elemental area, according to the example of Figure 9b.

E. Sampling.

Sample at each non-adjusted traverse point for the time interval specified in the method being used (e.g., Method 5). If two successive traverse points have been relocated to a single "adjusted" traverse point, sample twice as long at the adjusted point as at non-adjusted points, taking twice as many readings, but record the data as though two separate points had been sampled, each for half of the total time interval. During the sample run, velocity head (ΔP) readings shall be taken at points downstream of, but directly in line with, the sampling points. The sampling rate through the nozzle shall be set based upon the ΔP readings; if a nomograph is used, be sure when setting it to use the correct value (~ 0.99) of the pitot tube coefficient.[7]

ALTERNATIVE SAMPLING STRATEGY (STEADY-FLOW ONLY)

If the average total volumetric flow rate in a duct is constant with time, it is unnecessary to monitor stack gas velocity during a sample run. Thus, whenever time-invariant flow is believed to exist in a stack (e.g., for a steady-state process), the following traverse procedures can be used in lieu of those outlined in the preceding sections:

A. Location of Sampling-Velocity Measurement Site.

When steady flow is believed to exist in a duct, the sample and velocity traverses can be conducted non-simultaneously; therefore, the sampling and velocity measurement sites need not be separate. Rather, a

Table 2. CROSS-SECTIONAL LAYOUT FOR RECTANGULAR STACKS

No. of traverse points	Layout
9	3 x 3
12	4 x 3
16	4 x 4
20	5 x 4
25	5 x 5
30	6 x 5
36	6 x 6
42	7 x 6
49	7 x 7

single location can be used for both sampling and velocity measurement (see Figure 10).

Select a sampling-velocity measurement site that is at least 8 duct diameters downstream and 2 diameters upstream from the nearest flow disturbances. For rectangular stacks, use an equivalent diameter (Equation 2) to determine the upstream and downstream distances. If a sampling-velocity measurement site located 8 diameters downstream and 2 diameters upstream from the nearest disturbances is not available, choose a site that meets these criteria as nearly as possible. Under no circumstances, however, should a sampling-velocity measurement site be chosen that is less than 2 diameters downstream and 0.5 diameter upstream from the nearest disturbances.

B. Number of Traverse Points.

The correct number of traverse points shall be determined from Figure 11. To use Figure 11, proceed as follows: first, determine the distances "A" and "B" and express each distance in terms of duct diameters; second, read from Figure 11 the number of traverse points corresponding to each of these distances; third, select the higher of these two numbers of traverse points, or a greater number, so that for circular ducts the number is a multiple of 4 and, for rectangular ducts, the number is one of those shown in Table 2.

C. Location of Traverse Points, Circular Cross Sections

For circular stacks, locate the traverse points on 2 perpendicular diameters, according to Table 1 and the example of Figure 9a. Any traverse point located less than 1/2 inch from the stack wall will be unacceptable

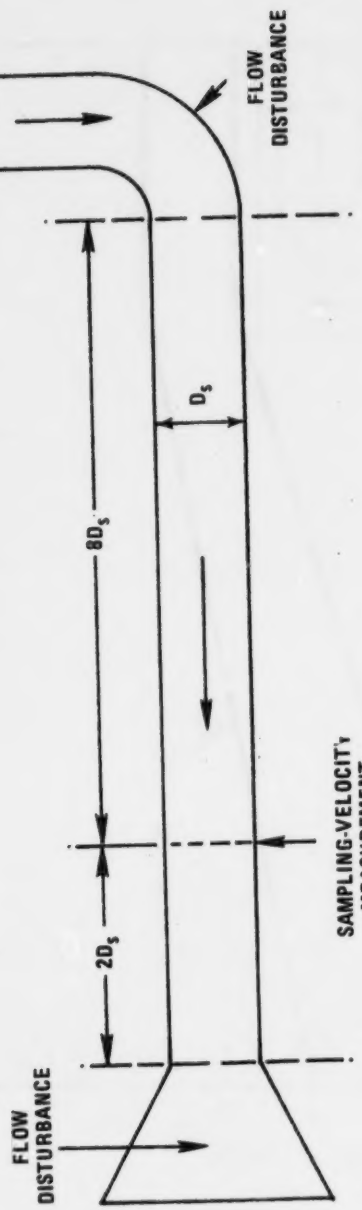


Figure 10. Recommended sampling arrangement; 4 in. $\leqslant D_s < 12$ in.; steady-flow only.

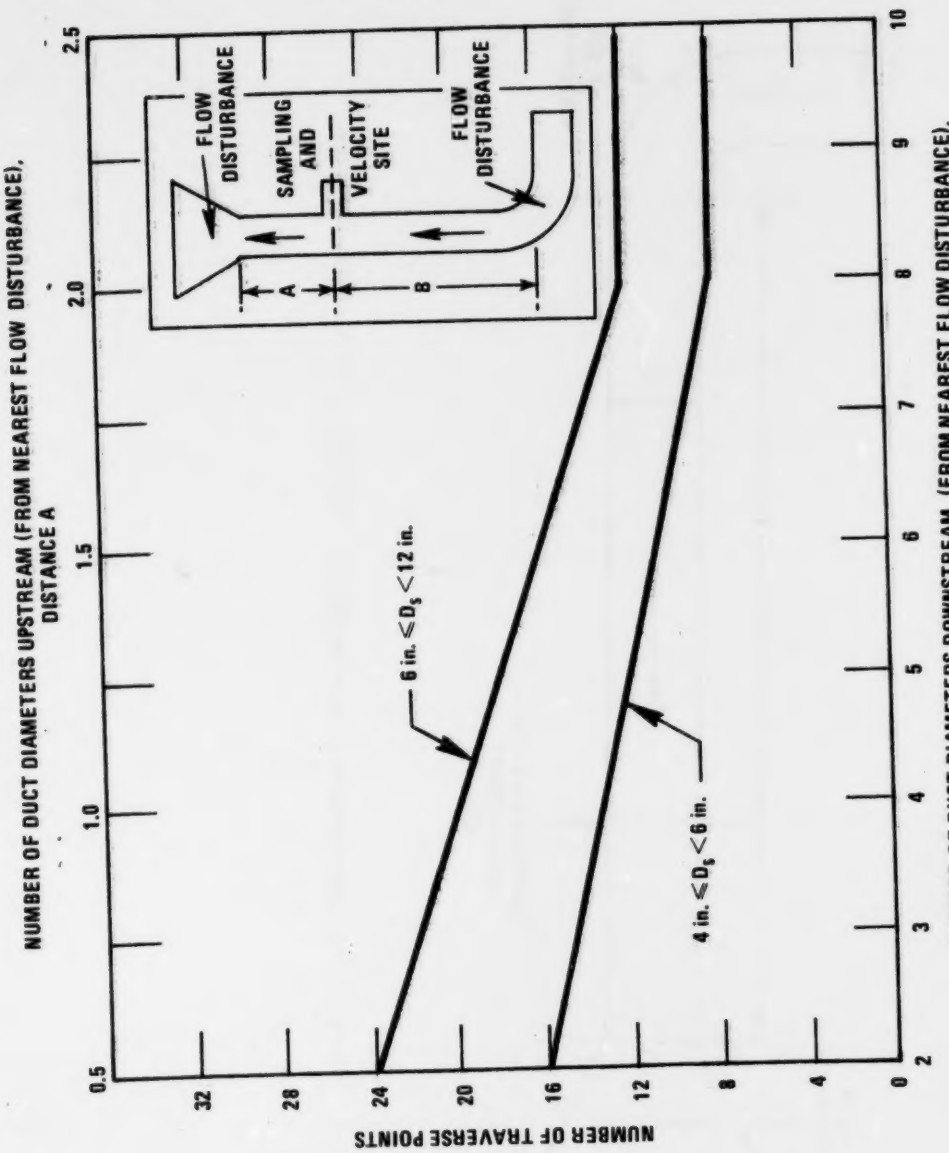


Figure 11. Minimum number of traverse points; 4 in. ≤ D_s < 12 in.; steady-flow only.

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GUIDELINES FOR SAMPLING IN TAPERED STACKS

T. J. Logan and R. T. Shigehara*

INTRODUCTION

Tapering of the inside diameter of stacks is occasionally done when designing natural draft stacks, when there are special flow or structural considerations, and for pressure recovery. These tapers seldom exceed a few degrees. Although guidelines for the selection of a sampling site to aid in the extraction of a representative sample are given in Method 1 of the December 23, 1971, Federal Register,[1] no mention is made about tapered stacks. The purpose of this paper is to provide the necessary background on how to deal with tapered stacks.

BASIC CONSIDERATIONS

In order to obtain a representative sample, the particles must be extracted at an isokinetic flow rate. The condition of isokineticity demands that the particles and gases flow directly into the sampling nozzle and that the velocity be accurately measured. Therefore, two factors must be considered: (1) the effect of the taper on flow conditions within the stack and (2) the effect of the taper on velocity determination and particulate matter collection.

Effect of Taper on Stack Flow Conditions

About the only information related to this area was the work done with venturi meters. The American Society of Mechanical Engineers research on fluid meters[2] indicates that beyond a convergent included angle of 21 degrees and a divergent included angle of 15 degrees, gas separation from the walls

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* Emission Measurement Branch, ESED, OAQPS, EPA, RTP, NC, November 1974

is expected to occur. This is undesirable as eddies would be formed, causing particles and gases to flow in undeterminable directions.

From a physical standpoint, convergent angles of 15 or 21 degrees would not likely occur in stacks due to the tremendous increase in velocity. If the larger stack diameter D is used, a tapered stack meeting the minimum 2.5 D requirement of Method 1 would cause an increase in velocity of about 8.6 times at the outlet for a 15-degree included angle and 186 times for the 21-degree included angle. Such an increase would require considerable additional power and would be impractical and uneconomical.

One builder of chimneys[3] related that convergent stacks generally do not exceed 0.5 in/ft. This corresponds to an included angle of about 4.8 degrees for convergent stacks. Divergent stacks are normally designed at about 5 to 15 degrees.

Based on the above, the 15-degree included angle can be considered the maximum limit for both convergent and divergent stacks, with the understanding that the 15-degree angle will be very unlikely in convergent stacks. The purpose for making this statement is to form the limit and basis for evaluating the effect of the taper on the velocity determination and the particulate matter collection.

Effect of 15-degree Included Angle on Velocity and Particulate Concentration

Convergent or divergent stacks with an included angle of 15 degrees would cause a maximum 7.5-degree angle of attack on the pitot tube and particulate sampling probe nozzle. Data presented by Grove and Smith[4] show that a 7.5-degree angle will result in velocity measurements with a type-S

pitot tube being biased 3.5 percent high. This higher apparent velocity also causes particulate sampling to be in error because isokinetic sampling requires that the sample gas velocity be made equal to the stack gas velocity, which is in error since it is measured by the misaligned pitot tube. In addition to the sampling rate being over-isokinetic, the misalignment of the probe nozzle with the stack gas stream results in a reduction of 0.85 percent in the effective nozzle area.

The magnitude of the effect on the particulate concentration by being over-isokinetic and having a reduced nozzle area is a function of particle size. For particles of less than 1 micrometer, the concentration will not be affected. However, with the larger particles of greater than 50 to 75 micrometers, the sampled concentration will be low; a bias of about 4.3 percent will occur (about 3.4 percent from being over-isokinetic and 0.86 percent from the reduced nozzle area). In a practical case, where there is a distribution of particle sizes, the error will be considerably less than the 4.3 percent, and for well-controlled sources where the majority of the particles are characteristically small (<2 micrometers), the error will be near zero.

For pollutant mass rates, the error of the higher measured volumetric flow rates will cancel out the errors of the lower measured concentrations, with the true concentration being between the maximum limits of +3.5 and -0.8 percent.

RECOMMENDATIONS

Based on the above discussion, the following guidelines, which should not

cause maximum errors greater than 4.3 percent in measured concentration or 3.5 percent in mass rate determinations, are recommended (actual errors for small particle sizes will be from 0 to -0.8 percent):

1. Consider all stacks with the total included angle of $\leq$15 degrees as straight stacks. If this angle is exceeded, consider the taper to be a flow disturbance and modify the stack with a straight section of at least 2.5 D.
2. Use the maximum diameter at point of upstream or downstream disturbance and Method 1 for determining the sampling point location and number of sampling points.

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CONSIDERATIONS FOR EVALUATING EQUIVALENT STACK SAMPLING TRAIN METERING SYSTEMS

R. T. Shigehara*

Introduction

The basic purpose of sampling train equipment is to collect a representative sample from a point (small area) within a stack cross-section or, when conducting a sample traverse, to collect a series of such samples. To accomplish this, the sampling train must (1) maintain either isokinetic or proportional sampling rate, depending on whether particulate or gaseous pollutants are being sampled, (2) efficiently collect reproducible samples of the pollutant at known levels, and (3) accurately measure the sample gas volume. Thus, conventional sampling trains incorporate some means of gas metering to regulate the sampling flow rate and to measure the sample gas volume.

"Method 5 - Determination of Particulate Emissions from Stationary Sources, Section 2.1.6" specifies the above requirements. It states, "Metering system - Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 5° F, dry gas meter with 2% accuracy, and related equipment, or equivalent, as required to maintain an isokinetic sampling rate and to determine sample volume."

There are many different workable metering techniques or systems. Individual stack samplers and control agencies usually have their own ideas as to which mode is the best. The purpose of this paper is to propose criteria to evaluate the different stack sampling train metering techniques or systems.

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Criteria for Isokinetic Sampling - EPA Particulate Test Train

The EPA particulate test train[1,2] will be used as a baseline reference for this discussion on the development of criteria for evaluating the different stack sampling train metering techniques or systems. The EPA train uses the pitot tube-orifice meter-dry gas meter system for setting isokinetic rates and for determining sample gas volume. In this system, the pitot tube is attached to the probe so that the gas velocity at each of the sampling points can be constantly monitored. The observed pitot tube manometer reading is related to the orifice meter manometer reading by an equation such that the flow rate through the sampling train can be adjusted to isokinetic conditions. To perform these calculations, the EPA train utilizes a nomograph, which requires as little as 5 to 10 seconds to determine and adjust the sampling rate after a new velocity reading or a change in stack flow has been observed. The nomograph is only a type of aid. Graphical techniques or electronic calculators can also be used to yield the same result. The dry gas meter is used to measure the sample gas volume and to measure the gas sampling flow rate independently from the orifice meter.

For particulates, it is the "condition" of isokineticity that ensures the extraction of a representative point sample, not the "means" by which the desired sampling rate is achieved. However, it is insufficient to simply state that all metering systems that have at some time demonstrated capability of obtaining isokinetic conditions are equivalent. All techniques, null balance, pitot tube-rate meter, pitot tube-volume meter-timer, and others, rely on the knowledge, experience, and conscientiousness of the

operator and require error-free equipment performance during sampling. Some assurance that isokinetic conditions were maintained throughout the actual sampling run is needed.

The EPA pitot tube-orifice meter-dry gas meter system provides sufficient proof of isokineticity:

1. All the components can be calibrated against a standard. The dry gas meter and orifice meter can be calibrated against a wet test meter (secondary standard) or a spirometer (primary standard). With periodical calibrations, the dry gas meter can maintain an accuracy of about 1% in volume measurement.[3,4] The type-S pitot tube can be calibrated against a standard type pitot tube which generally has a calibration coefficient between 0.98 and 1.00.[5] If one would send a standard pitot tube to the National Bureau of Standards, a certified calibration for velocity ranges from 6 to 100 fps or 6 to 155 fps can be obtained.

2. The pitot tube attached to the probe allows the velocity to be monitored and isokinetic sampling to be maintained throughout the entire test run. In this manner the sampling can be conducted under normal everyday process conditions; it is not limited to steady-state conditions of gas velocity.

Some limitations of the pitot tube for monitoring and measuring velocity should be recognized: (1) It still relies on the operator to properly orient the pitot tube into the direction of flow, correctly set up the manometer, and accurately read the velocity pressure head; (2) the pitot tube also has a lower velocity limit, usually reported at about 10 fps. This limitation is caused mainly by the difficulty in reading the manometer

scale; (3) in addition, the pitot tube is dependent on the density of the flowing gas stream. Thus, "great" changes in temperature, pressure, and gas composition (particularly moisture) may cause difficulty in determining the gas velocity and in setting isokinetic rates. Shigehara et al.[6] show a method of analysis to determine how much variation in the parameters can occur before "significant" errors result.

One method of reducing the problem of setting isokinetic rates when the gas density or composition of the stack effluent changes "significantly" with time is to place the orifice meter immediately after the filter, which is heated to stack temperature and does not allow moisture to condense. This method eliminates the problem of changes in composition, but adds the variable of total pressure at the orifice. However, this does not solve the problem of determining velocity.

The problems of low velocities and great changes in gas density have not yet been adequately solved. Until better means are specified, we can only attempt to increase the sensitivity of the manometer for low velocities and for great changes in gas density, to evaluate the source conditions and use techniques that, in our opinion, would provide adequate results.

3. The pitot tube-dry gas meter combination allows an overall average and individual average point deviations from isokinetic conditions to be calculated for each test run. This is helpful in that it permits acceptance or rejection of a run based on per cent of isokineticity actually obtained. The Federal Register[1] allows an overall average deviation of 10% from isokinetic. Smith et al.[7] have shown a calculation

method by which a deviation of 20% can be tolerated with the assurance that the sample concentration will be within 10% of the true concentration.

4. The orifice meter-dry gas meter combination provides a cross-check of flow rate and sample gas volume.

However, although both the dry gas meter and the orifice meter can yield accurate results, there is no means for checking against improper use or malfunctions under the actual operating conditions of the sampling train if the components are used separately.

Summarizing, it is the condition of isokineticity that produces a representative point sample. Any means that provides this condition could theoretically be considered equivalent. However, as improper uses or errors do occur, "sufficient proof" can be defined as:

1. All components be calibrated against a standard.
2. Velocity be monitored constantly and simultaneously with sampling.
3. A check of isokineticity actually obtained be provided.

Null Balance Probe System

This system is deceptively simple in principle. Also called static balance, zero pressure, and isokinetic probes, the pressure null balance probe is a nozzle specifically designed to measure the static pressure of the stack gases flowing around and within the probe nozzle. When both static pressures are equal, isokinetic conditions are said to exist. Cooper[8] summarized as follows:

"However, numerous problems have been observed in attempting to accurately maintain true isokinetic sampling conditions because the existence of equal pressures at outer and inner probe walls does not necessarily mean that equal velocities exist at both points. Differences in frictional flow losses between inner and outer surfaces caused by turbulence and surface nonuniformities, progressive coating and possible plugging of the inner static tap by particles, and possible differences of static tap location may all produce these conditions. Parker ("Some Factors Governing the Design of Probes for Sampling in Particle- and Drop-Laden Streams," Atmospheric Environment 2:477-490, September 1968) found that null balance systems had limited usage for large probes greater than 3/4 inch diameter. Toynbee and Parkes ("Isokinetic Sampler for Dust Laden Gases," International Journal of Air and Water Pollution 6:113-120, 1962) postulated that by a slight expansion of the rear section of the probe the inner frictional losses could be reduced inside the nozzle, and the system could be used over the velocity range from 600 to 2500 fpm. However, subsequent comments by Nonhebel in the same issue stated that the plugging problems associated with the inner static taps could not be overcome. Work by Dennis ("Isokinetic Sampling Probes," Industrial and Engineering Chemistry 49:294-302, 1957) and Hemeon and Haines ("The Magnitude of Errors in Stack Dust Sampling," Air Repair 4:159-164, November 1954) indicated that it was not always possible to assure isokinetic sampling conditions, and found the errors at different velocities for two nozzle sizes when departing from nozzle conditions."

This excerpt illustrates that even with careful calibration for the specific source and conditions, one cannot be positive that isokinetic conditions existed throughout the entire run. Although the feasibility of such a system has been demonstrated under controlled conditions, it suffers from the lack of proof of isokineticity for the actual operating conditions as provided by the EPA metering system. In order to provide sufficient proof of isokineticity, the null balance probe system must incorporate a pitot tube and a dry gas meter. This is what Wilson and Falgout[9] did to show that their null probe design was workable.

Dry Gas Meter as a Rate Measurement Device

The volume meter (dry gas meter), in addition to measuring the total sample volume, could serve as a rate meter for setting isokinetic rate by timing the needle travel. However, since the needle travel must be observed for one or more whole revolutions to obtain a reasonably accurate rate value, the rate is only an average, and changes are possibly delayed one or more minutes past the time they occur. Thus, its application is limited to sources where velocity is "fairly" constant. There is also the disadvantage of not having a cross check of volume and rate under actual operating conditions as with the orifice meter-dry gas meter combination.

Proportional Sampling

The same criteria apply to proportional sampling as to isokinetic sampling. It is the condition of proportionality that counts, not the means by which proportional sampling is achieved.

Like the EPA particulate sampling train, the same pitot tube-orifice meter-dry gas meter system can be used to regulate and check proportionality. But because of the lower sampling rates used for gaseous trains, a rotameter is normally used instead of an orifice meter.

Total Gas Sample Volume

The usual means for measuring the gas sample volume are dry gas meters, or rate meters such as orifices and rotameters. Cyclones, venturi meters, evacuated containers, critical orifices, and mass flow rates are also used. Whatever the means, it is the total gas volume that is desired. Integrating volume meters such as the dry gas meters, when sized properly, readily provide the desired result.[10] As mentioned previously, the dry gas meter can maintain an accuracy of about 1% in volume measurement when calibrated periodically against a wet test meter or spirometer.[3,4]

Rate meters can also be used to measure the sample gas volume. However, they measure instantaneous flow, which is subject to density changes of the gas stream. Therefore, other variables such as time, temperature, pressure, and pressure drop must be carefully recorded during the test run so that an integrated total volume can be calculated or obtained graphically. The same is true with dry gas meters if they are placed before the pump, because pressure could vary considerably, at times, during the test run as particulate matter builds up on the filter material.

The EPA sampling train places the dry gas meter and orifice meter behind the gas pump with the orifice meter open to the atmosphere. There are several practical advantages with this placement, which requires that the pump be leak proof. The advantages are:

1. The dry gas meter is subjected to a fairly constant pressure--the only variation coming from the orifice meter pressure drop, which is no more than 10 in. of water. The orifice meter is at a relatively constant atmospheric pressure; therefore, there is no need to record or to observe for all practical purposes pressure and meter readings extra carefully.

2. The dry gas meter need not be calibrated under the expected range of negative pressures that would occur if it were placed before the pump to compensate for the leakage around the meter diaphragm valves, particularly under high vacuums.

3. It is not necessary to have special gas meters that can withstand the high vacuums.

Condensers

Condensers are generally an integral part of a metering system. Their main purpose is to prevent moisture from condensing within the pump and gas metering devices. They also serve as a means for the determination of the average moisture content over the sampling duration.

The EPA test method gives a clear procedure for determining moisture when the gas stream does not contain water droplets. (If liquid droplets are present, the gas stream is assumed to be saturated). The probe and filter holder are heated to a minimum of 225°F so that moisture contained

in the sample will remain in gaseous form until the gas has passed the filter. Following the filter is a series of four Greenburg-Smith impingers which are immersed in an ice bath. The first two impingers each contain 100 ml of water. This chilled water acts to condense and trap the water vapor contained in the hot gases coming from the filter holder. The third impinger is empty and acts as a trap to collect any entrained water which might be carried over from the first two impingers. Finally, the fourth impinger contains approximately 200 grams of silica gel. The silica gel adsorbs most of the moisture which remains in the gas stream; for a 1-hour sampling run, less than 3% passes through if the temperature at the third impinger is kept below 70°F and less than 15 in. Hg vacuum.[11] The water collected in the first three impingers is easily measured volumetrically, and the weight change in the silica gel gives the amount of moisture collected there. The amount of moisture in the gas stream thus measured, and the sample gas volume as measured by the dry gas meter are then used to determine the moisture content.

The choice of equipment is not important as long as the moisture collected and leaving the condenser and gas sample volume can be measured accurately. For long sampling runs (3 to 4 hours), condensation coils may be better than or as effective as the EPA method. Temperature and pressure must be measured at the exit of the condenser to account for the moisture still remaining in the gas stream. However, because at 10 in. Hg. vacuum and 70°F, the amount of moisture at saturation conditions is about 3.7% by volume, the silica gel should still be used to protect the pump and metering devices.

Summary

Flow rate regulation and sample volume systems have been discussed. The basic purpose of these systems is to ensure that a representative point sample is collected and that the sample gas volume is accurately measured. However, representativeness is not a direct measurement. Thus, individual measurements that ensure representativeness must be compared against a standard. In the absence of any standard, the question of which result is right when two sampling trains yield different values can never be answered. When a standard is not available and if an evaluation is desired, design and/or performance criteria which have been scientifically or arbitrarily derived must be used.

Since it is the condition of isokineticity or proportionality that is important in the extraction of representative point samples, any technique that provides these conditions can be used. However, since they are a vital part of obtaining representative samples, checks under actual operating conditions must be provided. In this regard, the pitot tube-rate meter-volume meter system offers clear advantages.

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Published in "Stack Sampling News," March 1975, Vol. 2, No. 9.

EVALUATION OF METERING SYSTEMS FOR GAS-SAMPLING TRAINS

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INTRODUCTION

In the December 23, 1971, Federal Register,[1] several types of gas-sampling trains are specified. Each uses basically the same types of components in its metering system, i.e. flow control valve, diaphragm pump, rotameter, and dry gas meter, but differs in the sequence in which they are arranged. The different sequences are summarized as follows:

1. Method 3 (Integrated Gas Sampling Train): Flow control valve, diaphragm pump, and rotameter. A flexible bag follows the rotameter in this train.
2. Method 4 (Moisture Sampling Train): Flow control valve, diaphragm pump, dry gas meter, and rotameter.
3. Method 6 (SO_2 Sampling Train): Diaphragm pump, flow control valve, rotameter, and dry gas meter.

A recent publication[2] reported an adverse effect on the calibration of dry gas meters in particulate sampling trains utilizing diaphragm pumps with bypass valve systems. Although the gaseous sampling train metering systems do not use a pump bypass valve, questions were raised on whether or not this same effect would also be present in the smaller gas-sampling trains. Thus, tests were conducted to determine the effect, if any, of the position of the control valve in relation to the pump and metering devices on the calibration of the dry gas meter.

During the course of the test program, certain problems with the leak check procedure and the diaphragm pump were encountered. The purpose of this paper is to report these findings and the results of this test.

PROCEDURE

Test Equipment

The test train components used were the same as those specified by Method 6, as published in the December 23, 1971, Federal Register. A wet test meter (0.05 ft^3/rev.) was connected to the inlet of the metering system. A drying tube was inserted immediately after the wet test meter to protect the rotameter, dry gas meter, and pump from moisture condensation. Schematics of the two sampling train arrangements used to determine the effect of valve position are shown in Figure 1.

Test Procedure

The test was conducted in the following manner:

1. A leak check was first conducted. This leak check consisted of plugging the inlet to the metering system (before the drying tube), leaving the control valve fully open, turning on the pump, and noting the travel of the dry gas meter dial. If any leaks were indicated, they were corrected before any test was conducted.
2. Using the rotameter as a flow rate indicator, the following information was gathered: rotameter reading, wet test meter reading and temperature, dry gas meter readings and temperature, barometric pressure, and running time. From the raw data, two values were computed: (1) the calibration factor (F), which is the ratio of dry gas meter volume to wet test meter volume, and (2) average standard flow rate (Q) obtained by dividing the wet test meter volume, after being corrected for moisture content, by the running time.

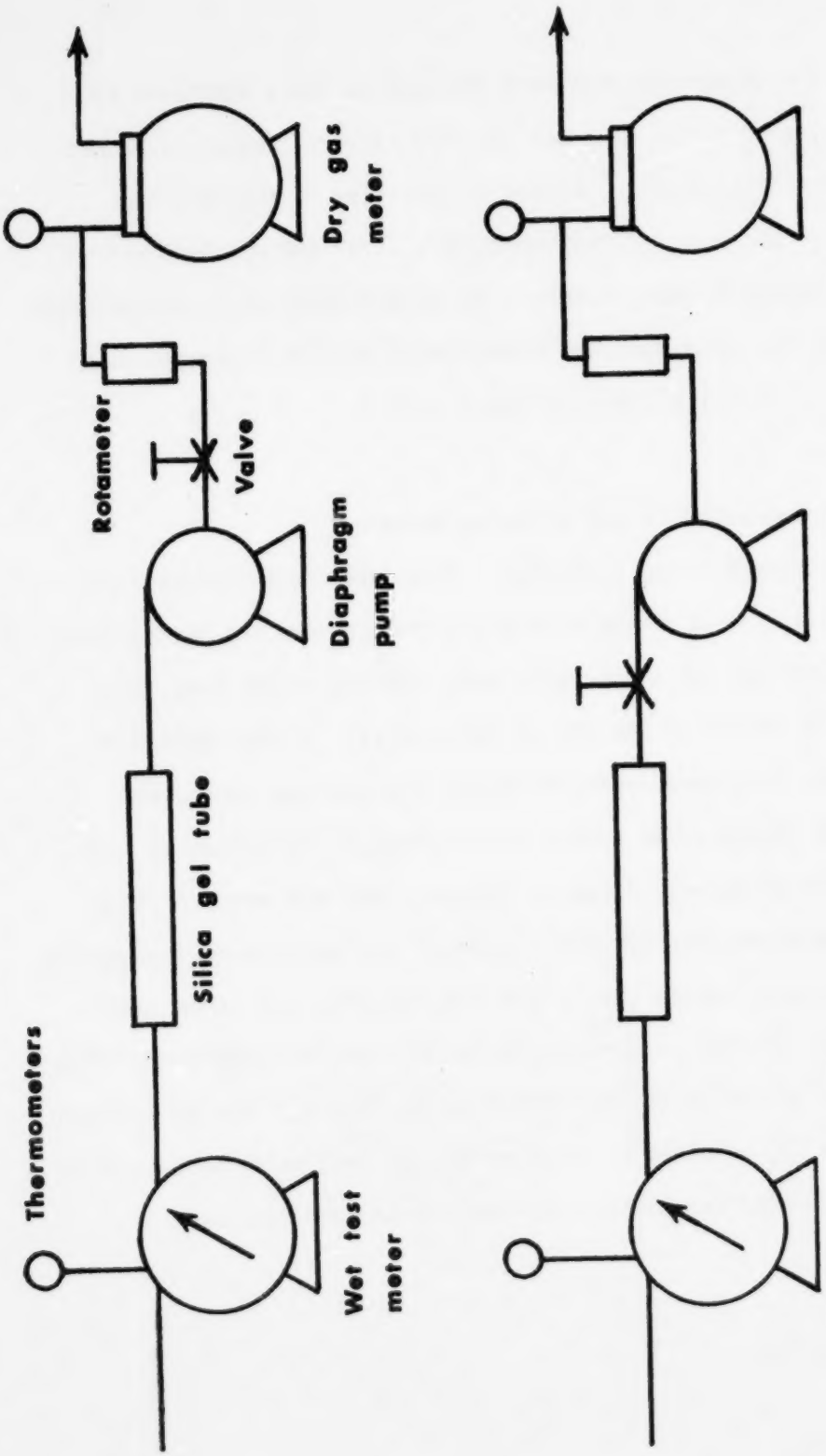

Figure 1

Initial test sampling train arrangements

TEST RESULTS

Four different sampling trains were tested, each with the valve before and after the pump. During one of the tests, with the valve placed after the pump and closed completely, movement of the wet test meter dial was noted. A leak check of the pump with a mercury manometer revealed a leak, which was not detected by the normal leak check procedure. (This leak was occurring where the diaphragm was connected by two screws to the connecting rod.) Plots of the calibration factor, F, versus the flow rate, Q, for pumps with and without leaks are shown in Figure 2.

During these tests, it was also noted that with the valve placed before the pump, the rotameter readings were greatly affected due to the pulsating motion of the diaphragm. But there was less of an effect on the calibration factor over a wider flow range with this arrangement than with the valve placed after the pump. The calibration factor was also less affected by leaks, when present, with this arrangement. Since these were desirable characteristics, steps were taken to reduce the effect of the pulsations. This was easily accomplished by placing a surge tank between the pump and the rotameter or by using the dry gas meter as a surge tank, i.e., interchanging the position of the dry gas meter and rotameter. The results are shown in Figure 3. Using the dry gas meter as the surge tank, however, caused the control response of the rotameter to be sluggish. Therefore, the surge tank placed before the rotameter was selected, and the final train shown in Figure 4 was used for subsequent tests.

After ensuring that all systems were leak free, this time using the manometer or the wet test meter procedure for the leak check, the tests were rerun. The results are shown in Figure 5.

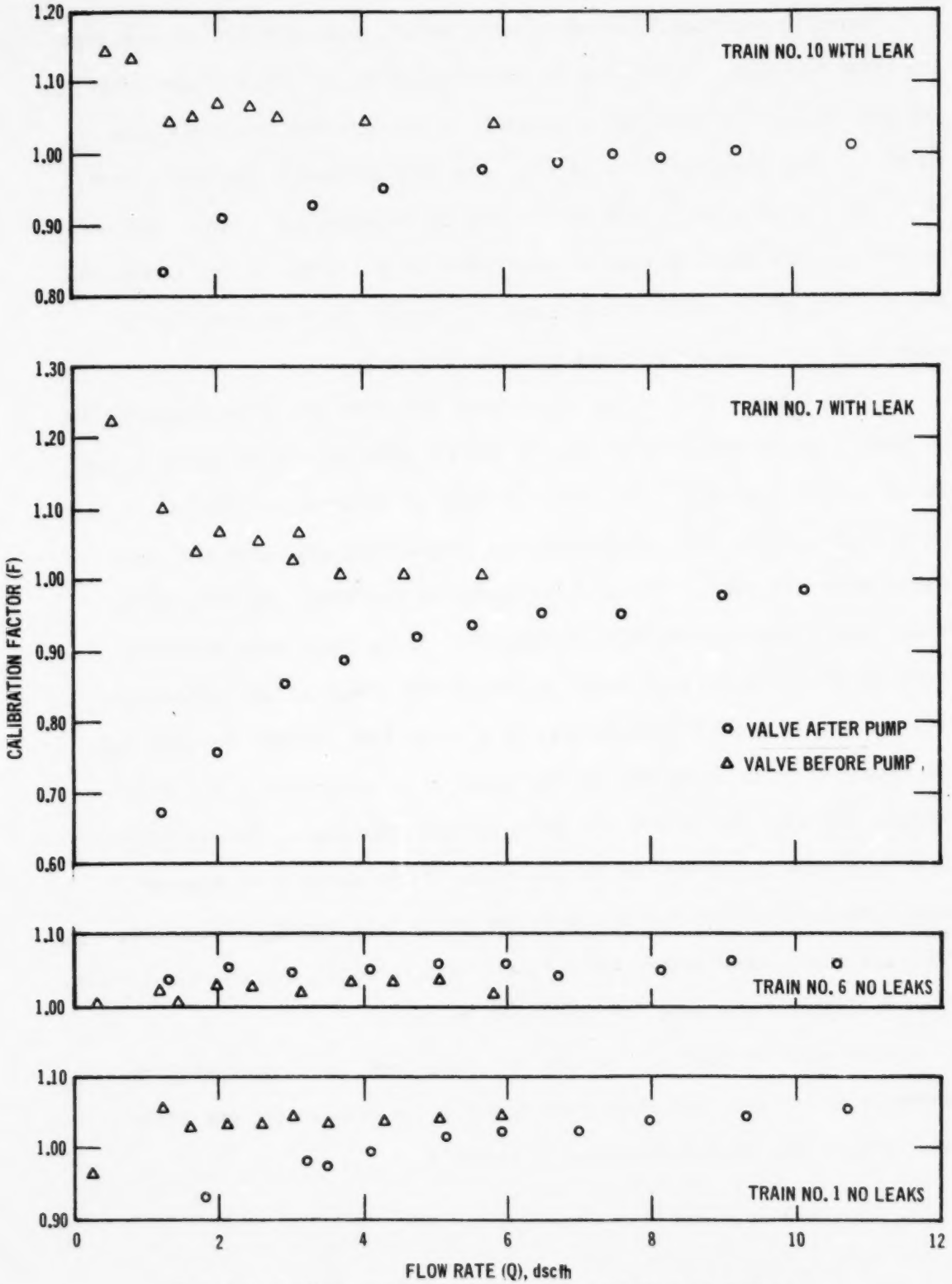

Figure 2. Calibration factor versus flow rate for Figure 1 Trains.

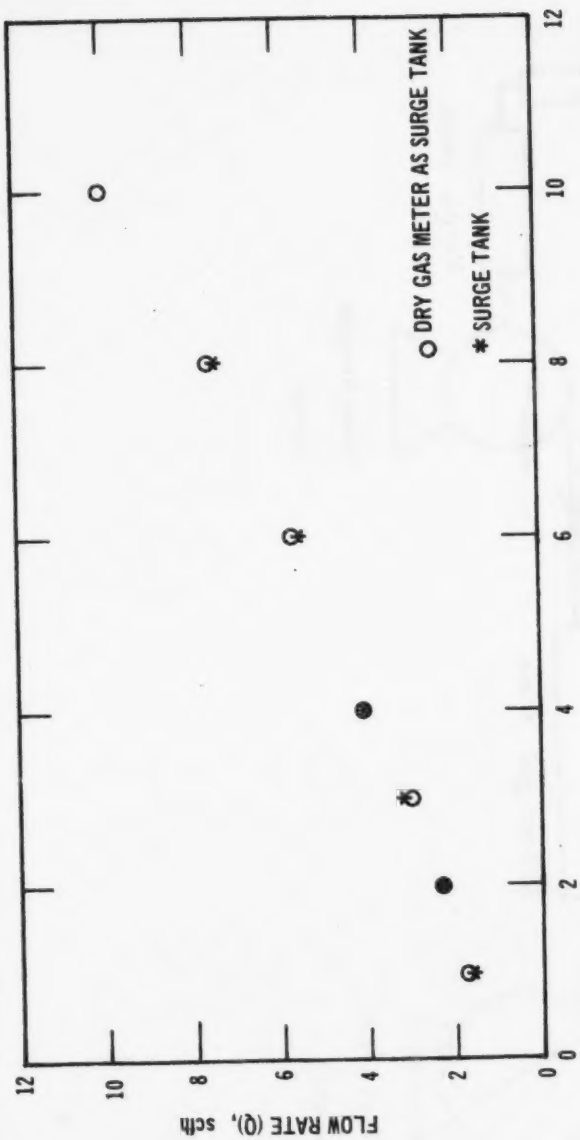

Figure 3. Rotameter calibration.

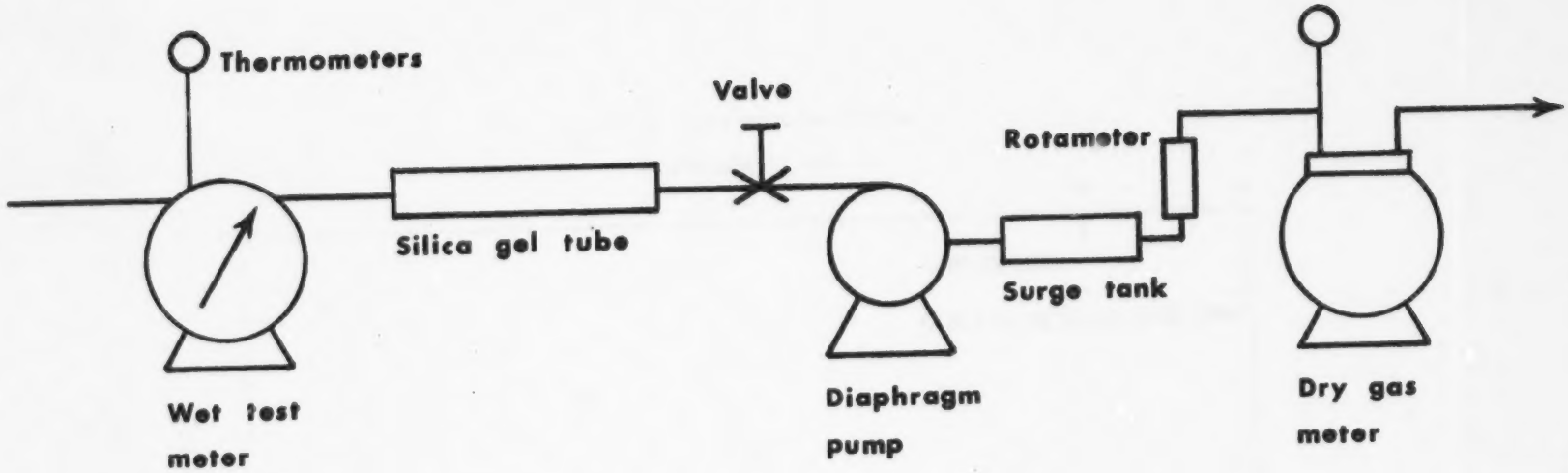

Figure 4

Final test sampling train arrangement

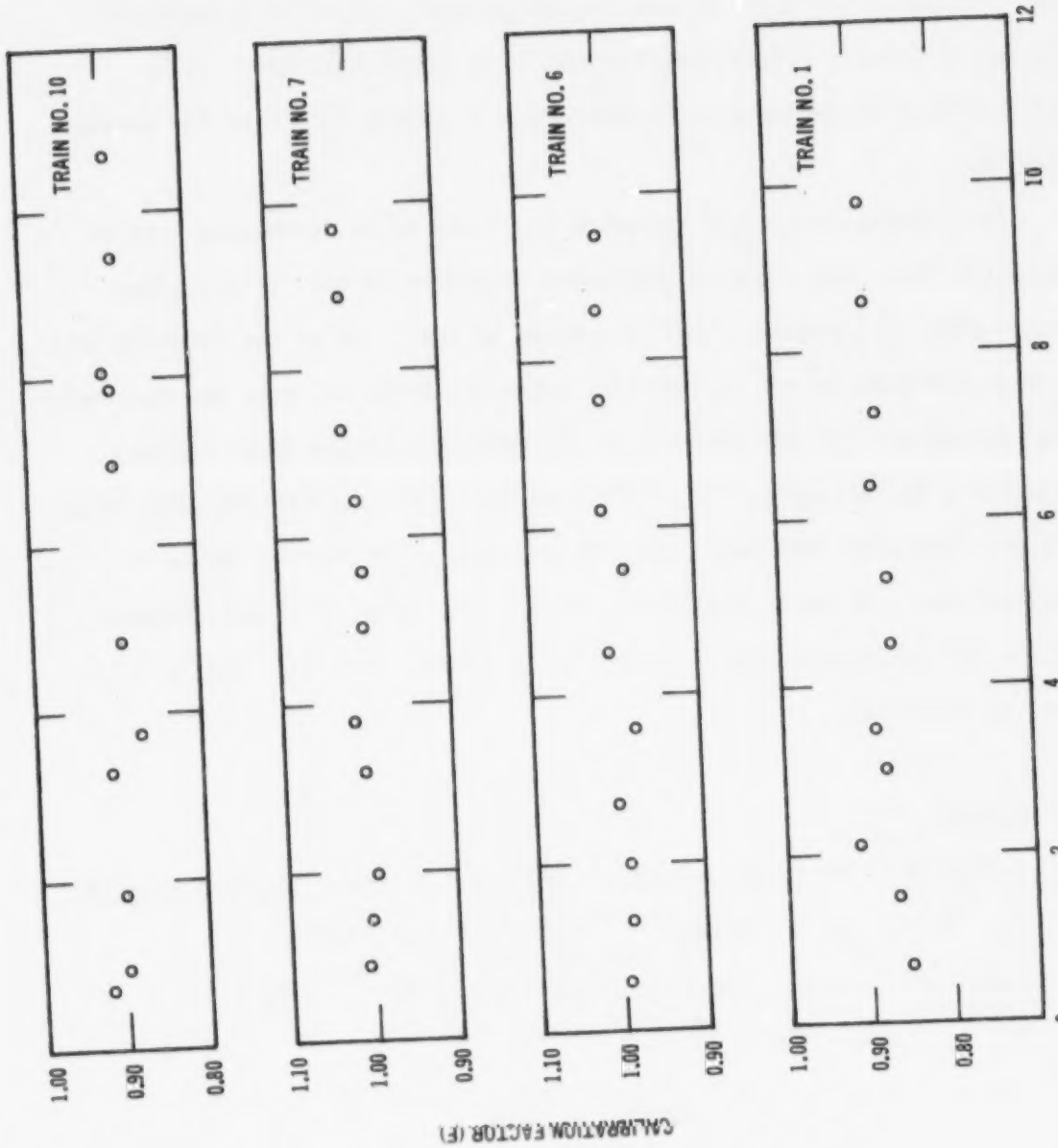

Figure 5. Calibration factor versus flow rate for Figure 4 trains.

SUMMARY AND RECOMMENDATIONS

The results of these tests showed that a constant dry gas meter calibration factor could be obtained whether the control valve was placed before or after the pump. However, the placement of the valve before the pump provided a constant calibration factor over a wider flow range and was not as greatly affected by leakages from within the pump. It is recommended that the metering system shown in Figure 4 be used for gaseous sampling.

The present leak check procedure was found to be inadequate. It is suggested that leak checks be conducted by either of the following two procedures: (1) connect a wet test meter at the inlet of the sampling train, turn on the pump, pinch off the line after the pump, and note wet test meter dial (suggested for laboratory), or (2) connect a vacuum gauge (mercury manometer, bourdon gauge, or similar) at the inlet, turn on the pump, pinch off the line after the pump, turn off the pump after maximum vacuum is reached, and note gauge reading (suggested for field use). Any movement of the wet test meter dial or vacuum gauge reading denotes a leak and must be corrected.

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AN EVALUATION OF THE CURRENT EPA METHOD 5 FILTRATION TEMPERATURE - CONTROL PROCEDURE

Robert F. Vollaro[a]

Introduction

Method 5, promulgated in the December 23, 1971 Federal Register[1], requires the use of probe and filter holder heating systems during isokinetic sampling. Prior to sampling, these heating systems are adjusted as follows: (1) the probe heater is set to provide a gas temperature of about 250°F* at the probe outlet; probe heater settings are obtained from Figure 21 of the sampling train operations manual[2], APTD-0576 (Figure 2 of this report); (2) the sample box thermostat is set to provide a temperature of approximately 250°F* around the filter holder. Although it is not explicitly stated in Method 5, one of the primary reasons for making these temperature adjustments is so that filtration will take place at 250°F ± 25°F*.

Recently, however, some observers have expressed concern over the adequacy of the above filtration temperature control procedure, particularly whether probe heater setting estimates made from the APTD-0576 reference curves will actually provide probe outlet temperatures around 250°F under field test conditions. Among the reasons given are: (1) the curves give no specific probe heater setting guidelines for sources with temperatures above 250°F or below 80°F; (2) the temperature of the gases surrounding a sample probe during an actual traverse will seldom be 80°F, which is the temperature base from which the curves are derived; and (3) the curves are strictly applicable only to gas streams of low moisture content. These comments fail to note that the

* Unless otherwise specified by a particular regulation.

[a] Emission Measurement Branch, ESED, OAQPS, EPA, RTP, NC, July 1975

reference curves were not originally intended to provide exact filtration temperature control; their original purpose was to furnish approximate guidelines by which moisture could be prevented from condensing ahead of the impingers. There is, nevertheless, question as to whether probe outlet temperatures around 250°F can be generated with confidence, even with the sample box set at 250°F.

In light of the above question, experiments were conducted, under a number of simulated field test conditions, to evaluate the present means of filtration temperature control. This paper reports the results of these experiments.

Experimental Set-up

The Method 5 sampling train configuration used in the experiments is shown in Figure 1. The components of the train met the design specifications outlined in the source sampling equipment construction manual, APTD-0581[3], except for the modifications necessary to facilitate temperature monitoring at the probe inlet, probe outlet, and inside the back half of the filter holder. Chromel-alumel thermocouples, insulated from the metal parts of the train, were used to monitor temperature in these experiments.

Filtration Temperature vs. Probe Outlet Temperature

Preliminary experiments were conducted to establish a relationship between probe outlet temperature and filtration temperature, at constant sample box setting. At each of three different box settings* (220, 240, and 260°F), the

* Note that the term "sample box setting," as used in this report, refers to the average temperature inside the box during a sample run. During sampling, the box temperature changed continually with time, rising and falling in 5-minute cycles between thermostatically controlled upper and lower limits.

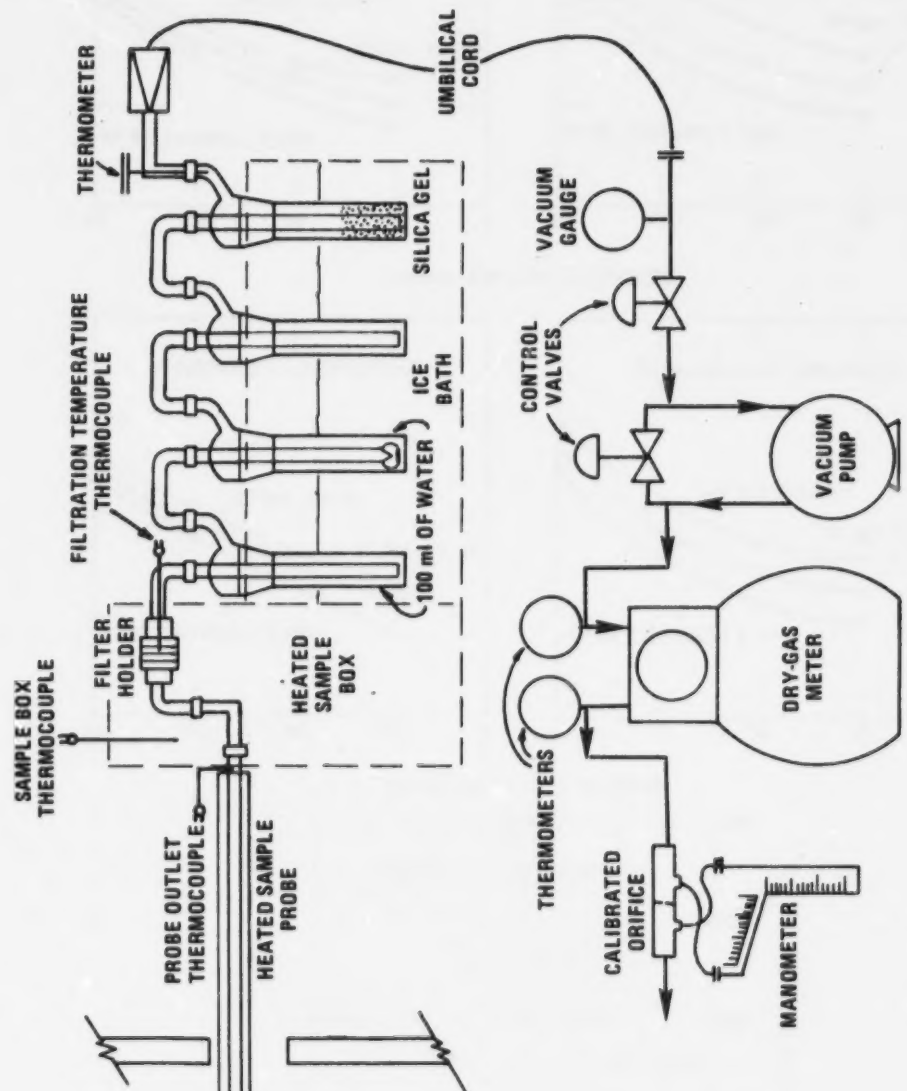

Figure 1. Sampling train configuration.

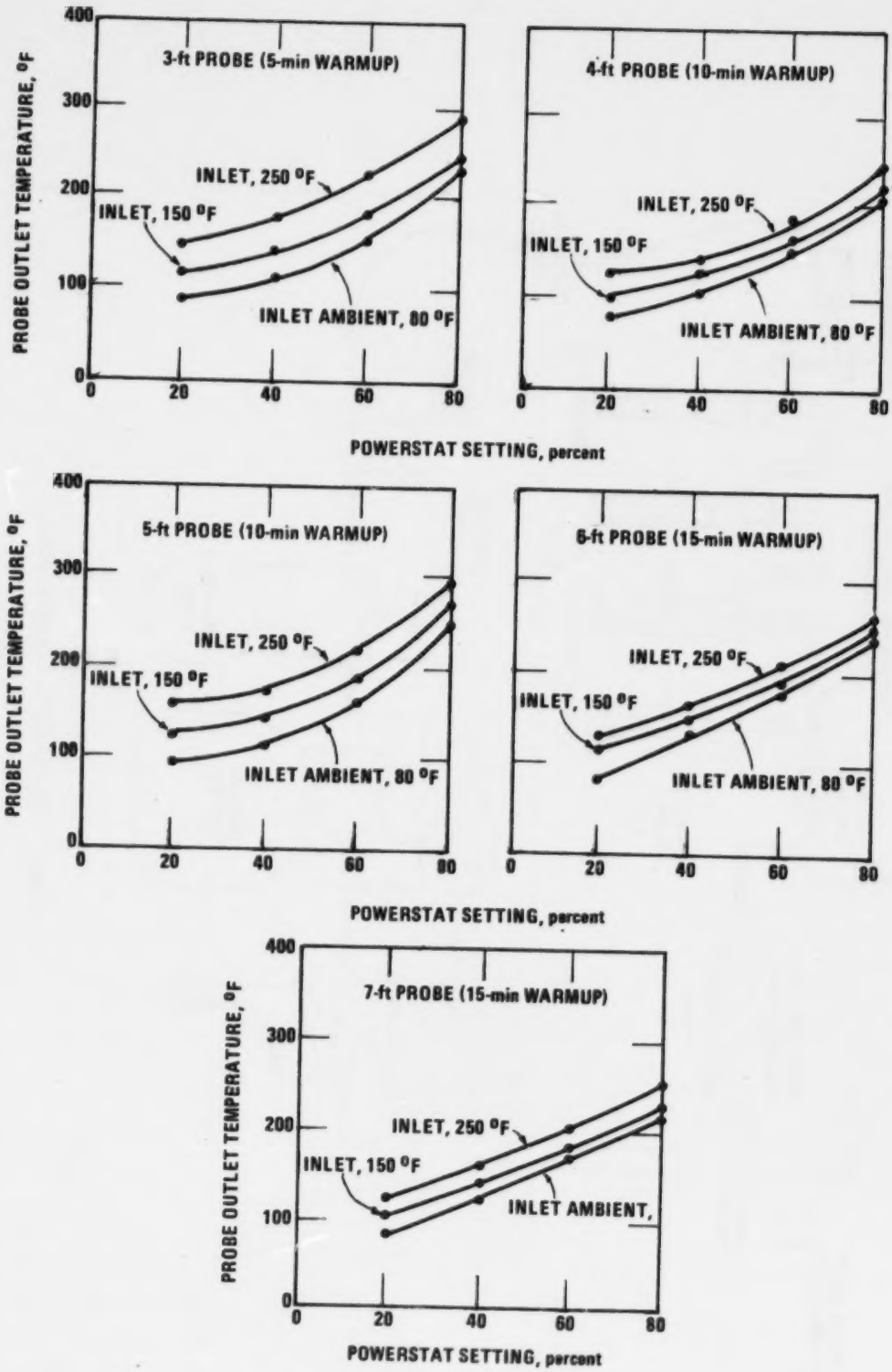

Figure 2. Probe temperatures.

temperature of the gas at the probe outlet was varied from 100 to 450°F while holding the sampling rate constant at 0.75 cfm. The results of these experiments are presented graphically in Figure 3.

Figure 3 shows that at constant sample box temperature, filtration temperature is a linear function of probe outlet temperature, requiring a 2.3°F change in probe outlet temperature to effect a 1°F change in filtration temperature.* Figure 3 also shows that, with the sample box set at its customary 250°F, it is necessary for the probe outlet temperature to be maintained between 230° and 350°F, in order for the filtration to take place at 250 ± 25°F.

Probe Outlet Temperatures

Further experiments were conducted, under a number of simulated field test conditions, to determine whether heater setting estimates made from the APTD-0576 curves (See Figure 2) would provide the necessary probe outlet temperatures to keep the filtration temperature between 225 and 275°F. Temperature was monitored during each run at the probe inlet, at the probe outlet, inside the sample box, and inside the filter holder, just behind the glass frit (See Figure 1). A constant sample rate of 0.75 cfm was maintained for all experiments. The following test cases were considered:

Test Case I--Possible Underheating. In this experiment, cold air at 37°F was drawn through a 3-foot sample probe. The sample box temperature was set at 260°F, and the temperature of the gases surrounding the probe was 37°F. In the absence of specific guidelines from APTD-0576 for probe inlet temperatures below 80°F, the probe heater was set according to the "closest available" probe

* This value will, of course, be a function of sample box design and the path length that the gas must go through (e.g., if a cyclone is used); however, a similar relationship should exist for different configurations.

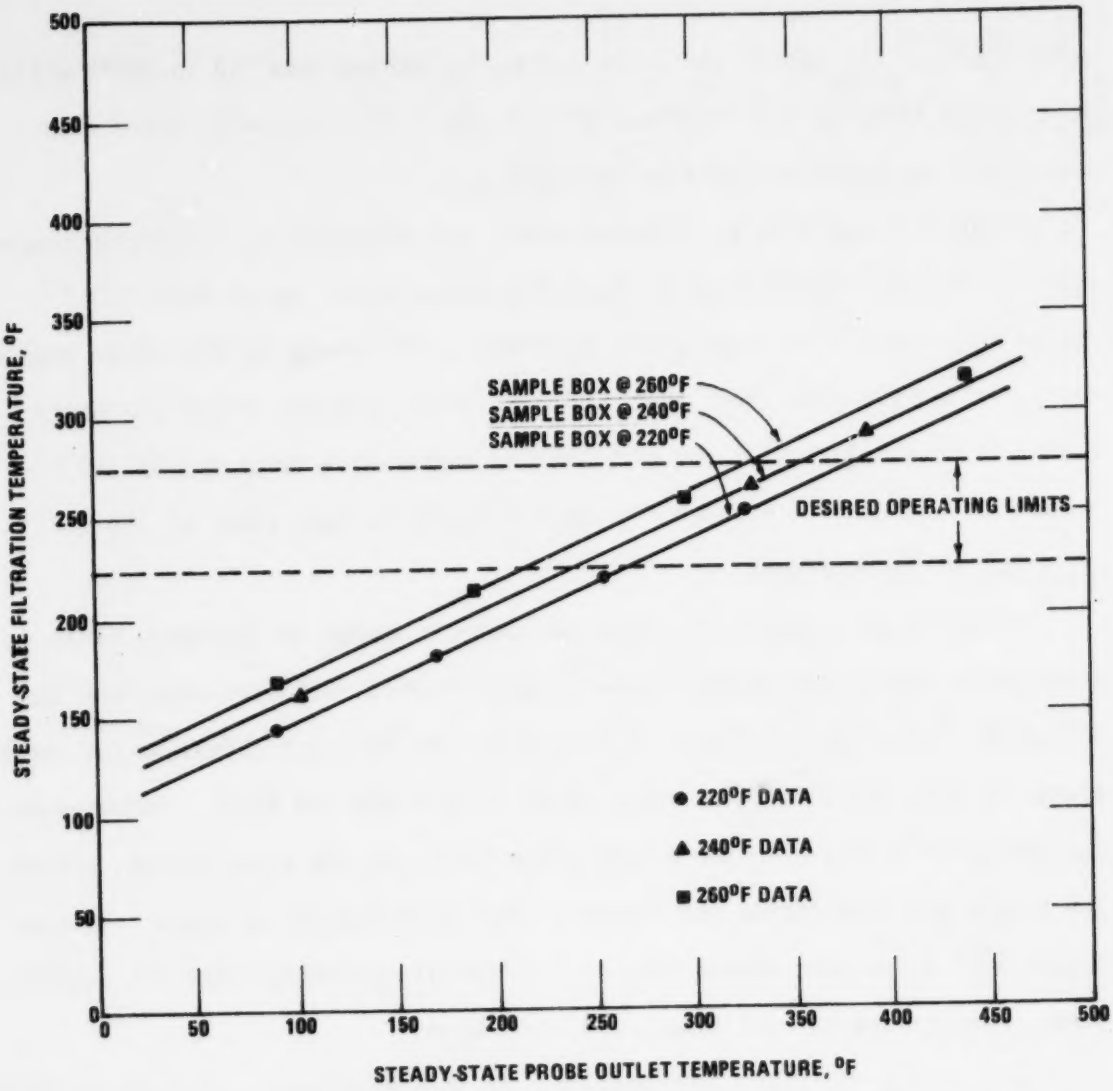

Figure 3. Filtration versus probe outlet temperatures (dry air).

inlet temperature curve, namely the 80°F curve. Case I was designed to simulate sampling from an ambient source with a short probe on a cold day. Its purpose was to determine if the probe heater was capable of heating cold sample gases, having only a short residence time in a probe set in cold surroundings, to an acceptable probe outlet temperture.

The results of this experiment are presented in the Appendix (See Table I). The data show that after a few minutes, the filter temperature had risen above 225°F; it continued to climb slightly thereafter, reaching a steady-state value of about 235°F. These results indicate that, even when a cold gas stream ($T<<80°F$) is sampled with a short probe set in cold surroundings, setting the probe heater by the appropriate 80°F inlet curve of APTD-0576 is satisfactory. Very little reduction in heater performance occurs, and a steady-state value of filter temperature safely within the range $250 \pm 25°F$ is rapidly established.

Test Case II--Possible Overcooling. Hot sample air at 475°F was drawn through an 8-foot probe set in 80°F surroundings; the sample box thermostat was set at 240°F. In the absence of APTD-0576 guidelines for sources hotter than 250°F, the probe heater powerstat was arbitrarily set at 25 percent. Case II was designed to simulate the testing of a very hot source ($T>300°F$) with a long sample probe. More specifically, Case II represents the outset of the sample traverse, when points close to the near stack wall are tested (i.e., when a good part of the probe is outside the stack), and overcooling of the sample gas can occur before it enters the filter box.

After a few minutes of the Case II sample run (See Table II in the Appendix), it was noted that the sample gases were cooling from 475°F at the probe inlet

to 175°F at the outlet. During this same time span, the filtration temperature reached only 190°F. For the remainder of the test, the probe heater setting was gradually increased, at 10-minute intervals, until filtration temperatures consistently above 225°F were obtained. When 225°F was reached, the powerstat setting was at 75 percent of maximum. Thus, the Case II data indicate the importance of proper probe heater calibration if the desired level of probe outlet temperature is to be achieved at the outset of the traverse of a very hot stack; random guessing at the powerstat setting to be used will not suffice.

Test Case III--Possible Overheating. A 20-inch diameter incinerator duct in which hot (520°F) combustion gases were flowing was sampled with a 3-foot probe. The probe was inserted as far as it would go into the duct, leaving about 16 inches of it exposed to the ambient (40°F) air. The sample box thermostat was set at 255°F. Again, in the absence of an APTD-0576 guideline, the probe heater was arbitrarily set at 20 percent. Case III was designed to simulate that stage of the sample traverse of a very hot (T>300°F) stack when points close to the far wall are tested and a good part of the probe is inside the stack, surrounded by hot gases. The purpose of this test was to check for possible overheating.

The Case III data (See Table III in Appendix) show that although the temperature was leveling out, overheating of the filter occurred after 16 minutes of sampling. After 17 minutes, the probe heater was shut off to try and bring the filtration temperature back below 275°F. The filtration temperature did drop to 274°F; however, had the ambient temperature been higher than 40°F or the

stack gas temperature higher than 520°F, overheating would most likely have continued, and to achieve lower temperatures an adjustment in the sample box temperature would have become necessary.

To determine the severity of filter overheating, had an arbitrary powerstat setting higher than 20 percent been chosen, Case III was repeated. This time, the probe heater setting was gradually increased, at 12-minute intervals, from 25 percent to 80 percent of maximum. These data are shown in Table IV (See Appendix). Filtration temperatures well in excess of 300°F occurred at the higher powerstat settings.

Test Case IV--Effect of Moisture. During Test Case III, when incinerator gases were sampled, the sample box was set at 255°F. However, a plot of filtration temperature versus probe outlet temperature (See Figure 4) produced a data line well above the 225°F region of Figure 3. It was assumed that the high moisture content of the combustion gases caused the difference. To check this assumption, four test runs were performed, in which moist air (estimated at 5 to 10 percent) at different temperatures (228, 270, 293, and 468°F) was sampled. The sample box was maintained at about 250°F.

The results of these tests are plotted in Figure 4. They confirm that moisture in the sample stream can alter the relationship between the probe outlet and filtration temperatures.

Conclusions

An evaluation of the present means of controlling filtration temperature in the EPA Method 5 train has demonstrated that:

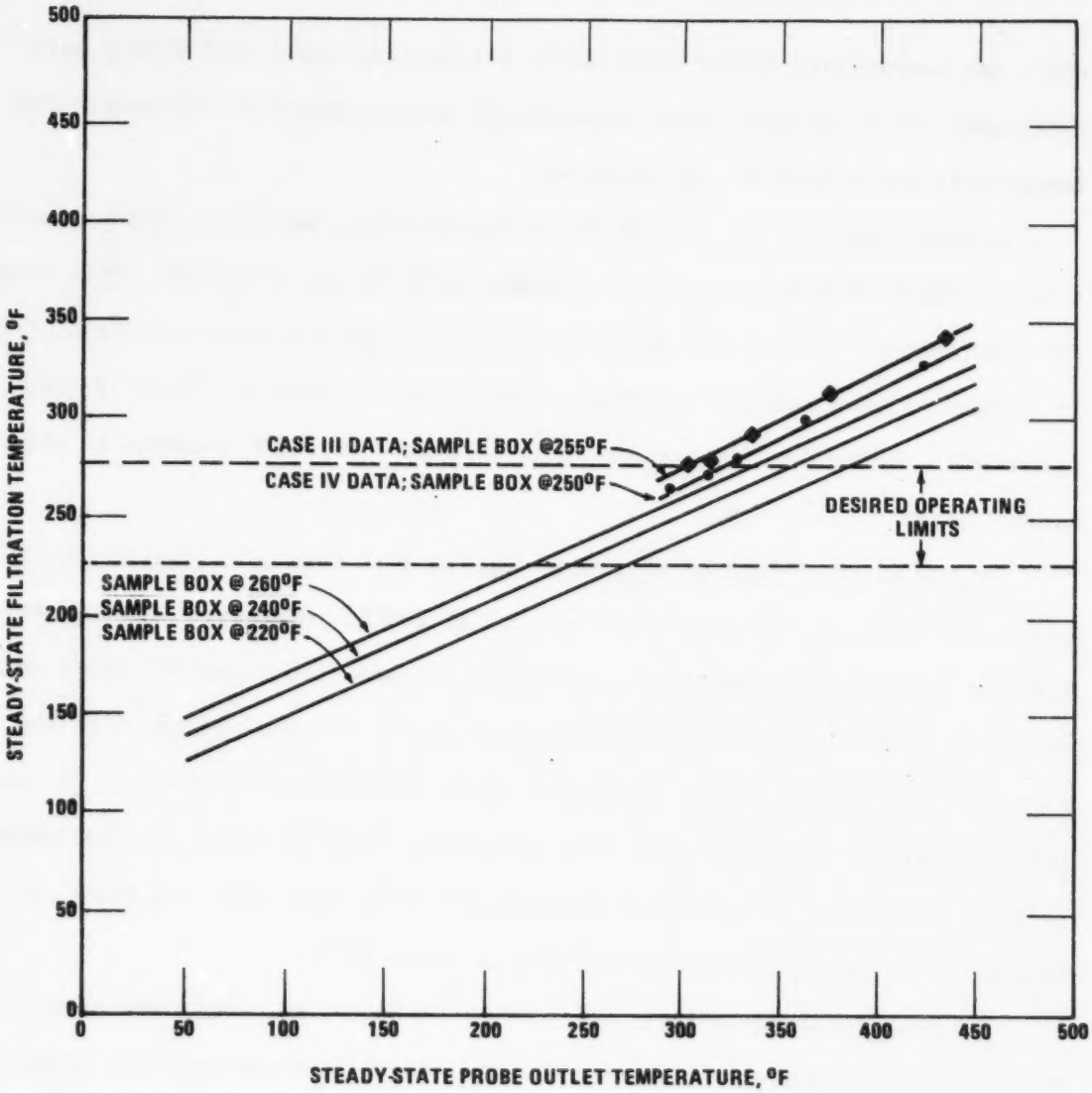

Figure 4. Filtration versus probe outlet temperatures.

1. At constant sample box setting, filtration temperature is a linear function of probe outlet temperature. High (>5 percent) sample-stream moisture content (or presence of water droplets) can, however, alter this relationship.

2. The APTD-0576 reference curves provide reasonable estimates of probe outlet temperature when applied to the sampling of streams similar to those upon which they are based, i.e., streams of low moisture content with temperatures between 80°F and 250°F. The use of the 80°F inlet curves for ambient streams with temperatures as low as 37°F has been shown to be satisfactory (Case I); by analogy, it can be inferred that the 250°F inlet curves will apply reasonably well to low-moisture streams with temperatures up to about 300°F.

3. For very hot stacks (T>300°F) and for stack gas streams of high moisture content (or containing water droplets), it has been demonstrated that the practical value of the APTD-0576 reference curves diminishes considerably. In these cases, sample gas overheating or overcooling at the probe outlet can occur (depending on the probe heater setting and the temperature of the gases surrounding the main body of the probe) and can cause the filtration temperature to be outside the desired operating limits.

REFERENCES

1. "Standards of Performance for New Stationary Sources," Federal Register, December 23, 1971.

2. Rom, Jerome, J.. Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment, Environmental Protection Agency. Publication No. APTD-0576. Research Triangle Park, N. C. 27711. March, 1972.

3. Martin, Robert M.. Construction Details of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Publication No. APTD-0581. Research Triangle Park, N. C. 27711. April, 1971.

APPENDIX

TABLE II: CASE II DATA

Date: 1/7/75

Case: II

Sample Rate: 0.75 cfm

Sample Box Setting: 240 °F

Probe Heater Setting: 25%

Operator: R. Vollaro

Time (Minutes)	Probe Inlet Temperature (°F)	Probe Outlet Temperature (°F)	Sample Box Temperature (°F)	Filtration Temperature (°F)	Powerstat Setting (%)
1	455	153	243	181	25
2	457	157	220	183	25
3	466	161	241	183	25
4	470	164	260	185	25
5	472	167	262	188	25
6	474	170	231	192	25
7	476	172	216	193	25
8	477	174	242	192	25
9	477	177	267	193	25
10	477	179	256	196	25
11	477	182	230	198	40
12	479	188	245	198	40
13	476	193	269	199	40
14	476	195	247	203	40
15	475	197	226	204	40
16	477	198	223	204	40
17	477	200	258	204	40
18	477	201	265	205	40
19	477	203	241	208	40
20	477	204	223	209	40
21	476	208	234	209	60
22	475	213	256	208	60
23	475	217	262	211	60
24	475	221	240	215	60
25	474	225	219	216	60

TABLE II

(Continued)

Time (Minutes)	Probe Inlet Temperature (°F)	Probe Outlet Temperature (°F)	Sample Box Temperature (°F)	Filtration Temperature (°F)	Powerstat Setting (%)
26	474	228	231	216	60
27	475	229	257	216	60
28	474	231	259	219	60
29	473	234	238	222	60
30	473	235	222	223	60
31	472	241	232	222	75
32	471	244	256	222	75
33	472	250	257	225	75
34	472	254	235	229	75
35	471	257	220	230	75
36	471	261	235	230	75
37	472	262	263	231	75
38	475	265	254	234	75
39	472	268	234	236	75
40	471	269	222	237	75

TABLE III: CASE IIIA DATA

Date: 12/6/74

Case: IIIA

Sample Rate: 0.75 cfm

Sample Box Setting: 255 °F

Probe Heater Setting: 20%

Operators: R. Vollaro and R. Mobley

Time (Minutes)	Probe Inlet Temperature (°F)	Probe Outlet Temperature (°F)	Sample Box Temperature (°F)	Filtration Temperature (°F)	Powerstat Setting (%)
1	508	259	272	223	20
2	509	272	269	235	20
3	510	280	245	245	20
4	513	286	225	252	20
5	514	291	236	255	20
6	511	296	266	257	20
7	513	299	273	262	20
8	514	302	247	266	20
9	516	304	226	267	20
10	515	305	253	267	20
11	514	306	276	269	20
12	517	307	283	272	20
13	519	309	256	275	20
14	521	310	234	275	20
15	522	311	251	275	20
16	524	314	277	275	20
17	529	314	274	276	20
18	534	313	249	278	0
19	535	309	228	278	0
20	532	306	235	276	0
21	532	305	265	274	0
22	533	304	282	274	0
23	532	304	256	275	0
24	533	304	235	276	0
25	535	304	236	274	0

TABLE IV: CASE IIIB DATA

Date: 1/27/75

Case: IIIB

Sample Rate: 0.75 cfm

Sample Box Setting: 255 °F

Probe Heater Setting: 25%

Operator: R. Vollaro

Time (Minutes)	Probe Inlet Temperature (°F)	Probe Outlet Temperature (°F)	Sample Box Temperature (°F)	Filtration Temperature (°F)	Powerstat Setting (%)
1	450	256	279	212	25
2	453	277	248	230	25
3	454	281	225	243	25
4	455	284	243	251	25
5	456	288	269	256	25
6	457	292	269	262	25
7	456	295	243	266	25
8	457	299	220	268	25
9	458	301	256	269	25
10	462	304	278	271	25
11	463	305	262	274	25
12	466	308	237	277	25
13	469	313	234	277	40
14	470	316	256	277	40
15	470	320	278	279	40
16	467	322	264	283	40
17	472	326	238	286	40
18	473	327	225	286	40
19	476	330	262	286	40
20	477	332	282	288	40
21	476	333	257	291	40
22	478	335	231	293	40
23	479	336	237	292	40
24	485	338	275	292	40
25	486	344	266	295	60

TABLE IV

(Continued)

Time (Minutes)	Probe Inlet Temperature (°F)	Probe Outlet Temperature (°F)	Sample Box Temperature (°F)	Filtration Temperature (°F)	Powerstat Setting (%)
26	493	352	244	298	60
27	490	357	224	300	60
28	490	361	248	301	60
29	492	365	275	304	60
30	491	367	273	307	60
31	492	371	247	310	60
32	492	372	227	312	60
33	491	373	253	312	60
34	492	375	273	312	60
35	496	376	274	315	60
36	497	379	247	316	60
37	500	395	228	317	80
38	502	404	261	319	80
39	503	413	286	325	80
40	503	417	269	328	80
41	509	424	243	333	80
42	506	428	224	335	80
43	508	431	256	337	80
44	507	432	275	338	80
45	511	434	283	339	80
46	511	435	269	341	80
47	511	436	251	343	80
48	509	438	227	344	80

LABORATORY EVALUATION OF SILICA GEL COLLECTION EFFICIENCY UNDER VARYING TEMPERATURE AND PRESSURE CONDITIONS

Peter R. Westlin and Fred C. Biddy*

Introduction

The impinger section of the EPA Method 5 sampling train[1] is intended to collect moisture from sample gases for determination of moisture content. The final stage of the collection train is an impinger with silica gel. Laboratory experiments were conducted in order to determine the effectiveness of the silica gel impinger as a moisture collector under various sampling conditions of temperature and pressure.

Equipment Set-up

Figure 1 shows the sampling train as it was used in the experiments. The moisture and heat source was a flask of water heated by a rheostat controlled hotplate. The sample entered the train through a flow control valve used to simulate resistance through the sampling train. The first and second impingers of the train were each filled with 100 milliliters (ml) of water. The third impinger was a dry impinger with wet-bulb and dry-bulb thermometers attached to the center tube. In order to reach the gas velocity necessary to obtain correct wet-bulb temperature readings, a 2.2-cm (0.87-in.) diameter orifice was placed in this impinger, and the thermometer tips were located in the orifice opening. The fourth impinger contained approximately 200 grams (g) of silica gel for each run. The silica gel was grade 42 and a 6-16 mesh size indicating type. Following the impinger section was another thermometer for measuring the temperature of the gas stream leaving the silica gel impinger. A standard EPA Method 5 meter box was used to draw and measure

* Emission Measurement Branch, ESED, OAQPS, EPA, RTP, NC, July 1975

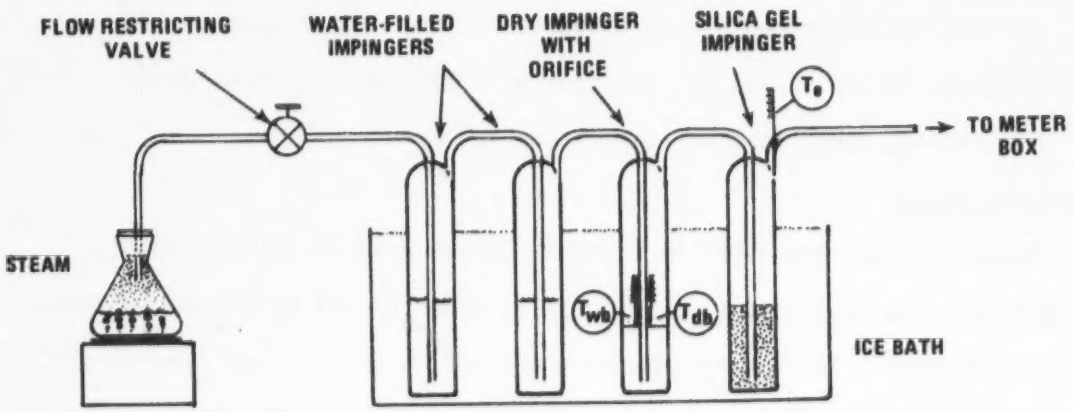

Figure 1. Laboratory moisture sampling train.

the volume of sample and measure the vacuum in the volume meter.

Procedure

At the beginning of the test run, the flow control valve and the meter box pump were adjusted to attain the desired meter vacuum and flow rate. Flow rate was maintained between 1.1 and 1.3 standard cubic meters per hour (scm/hr)(0.52 to 0.61 scf/min). Temperature in the impingers was controlled and maintained with an ice bath. Readings of meter volume, meter temperatures, train vacuum, wet-bulb and dry-bulb impinger temperatures, and exit-gas temperature were recorded at 5-minute intervals during the 2-hour runs. Adjustments to flow rate and train vacuum were made as necessary. The vacuum in the third impinger was determined so that the moisture content of gas entering the silica gel impinger could be calculated. This sample vacuum, reported in Table 1, was varied from 107 to 460 millimeters of mercury (mm Hg)(4.2 to 18.1 in. Hg).

The moisture entering the silica gel impinger, the moisture entering the meter box, and the moisture collection efficiency of the silica gel were calculated as follows:

1. Moisture fraction entering silica gel:

$$B_{wi} = \frac{P_s - 0.00066(1 + 0.00116b\ T_{wb})(P_i)(T_{db} - T_{wb})}{P_i}$$

Where:

B_{wi} = volume fraction of moisture, %/100

Table 1. MEASURED AND CALCULATED VALUES RELATIVE TO MOISTURE COLLECTED IN SILICA GEL IMPINGER IN EPA METHOD 5 SAMPLING TRAIN DURING LABORATORY TESTS[a]

Run	$P_a - P_i$ Sample vacuum, mm Hg	T_{db} Inlet temp., °C	T_e Exit temp., °C	V_m Meter volume scm	e_{sg} Collected H_2O vol., scm	$V_m + e_{sg}$ Total volume, scm	$B_{wi} \times 100$ Inlet H_2O, %	e_i Inlet H_2O vol., scm	e_e Exit H_2O vol., scm	$\frac{e_e}{V_m}$ Exit H_2O, %	$100 \times \frac{e_{sg}}{e_i}$ Collection efficiency, %
A	107	9.2	14.2	2.490	0.024	2.51	1.3	0.032	0.007	0.3	78
B	109	15.2	18.5	2.257	0.034	2.29	2.0	0.046	0.012	0.5	74
C	107	26.6	30.0	2.458	0.068	2.53	4.0	0.102	0.034	1.3	67
D	107	21.6	24.1	2.494	0.052	2.55	3.0	0.076	0.024	0.9	68
E	107	32.6	34.9	2.491	0.063	2.55	5.7	0.145	0.082	3.2	43
F	224	9.9	14.5	2.671	0.035	2.71	1.7	0.047	0.012	0.4	75
G	226	19.3	25.0	2.496	0.059	2.56	3.2	0.081	0.022	0.9	73
H	226	33.8	35.2	2.470	0.076	2.55	7.5	0.190	0.114	4.5	40
I	460	9.7	16.8	2.520	0.037	2.56	3.0	0.078	0.041	1.6	48
J	460	21.2	27.0	2.546	0.060	2.61	6.4	0.167	0.106	4.1	36
K	460	33.7	35.8	2.536	0.065	2.60	13.2	0.343	0.278	10.7	19

[a] Symbols above columns refer to calculations section of text.

P_s = saturated vapor pressure at T_{wb}, mm Hg

T_{wb} = wet-bulb temperature, °C

T_{db} = dry-bulb temperature, °C

P_i = absolute impinger pressure, mm Hg

2. Volume of moisture collected by silica gel:

$$e_{sg} = 1.342 \times 10^{-3} (M_{sg})$$

Where:

e_{sg} = moisture gas volume in silica gel, scm

M_{sg} = mass of water collected in silica gel, g

Note: Standard temperature and pressure are 21°C (70°F) and 760 mm Hg (29.92 in. Hg)

3. Total moisture volume entering silica gel:

$$e_i = B_{wi}(e_{sg} + V_m)$$

Where:

e_i = moisture volume entering silica gel, scm

V_m = standard dry-gas meter volume, scm

4. Collection efficiency of silica gel:

$$E = 100\left(\frac{e_{sg}}{e_i}\right)$$

Where:

E = collection efficiency, %

5. Moisture exiting the silica gel impinger:

$$e_e = e_i - e_{sg}$$

Where:

e_e = moisture volume exiting silica gel, scm

Discussion of Results

Table 1 shows the results of 11 test runs at three different train vacuums. Note that the calculated values represent averages over each of the complete 2-hour runs. As can be seen from this table, the moisture collection efficiency of the silica gel decreased as the inlet temperature and the exit-gas temperature increased. A result of this relationship is that the percent of moisture in the sample gas entering the meter box increased from 0.3 percent at 14.2°C (58°F) exit temperature to 3.2 percent at 34.9°C (95°F) exit temperature at the same train vacuum of 107 mm Hg (4.2 in. Hg).

Also shown in these results is the effect of sample train vacuum on collection efficiency. For example, looking at runs A, F, and I, the inlet temperatures are approximately equal at 9.5°C (49°F) while the train vacuum varies from 107 mm Hg (4.2 in. Hg) to 460 mm Hg (18.1 in. Hg). The silica gel moisture collection efficiency decreases from 78 percent at 107 mm Hg (4.2 in. Hg) to 48 percent at 460 mm Hg (18.1 in. Hg). Moreover, the amount of moisture exiting the silica gel increases from 0.3 percent to 1.6 percent over the same conditions. Figure 2 shows graphically the effect of both exit temperature and sample vacuum on the moisture content in the exit gas from the silica gel impinger.

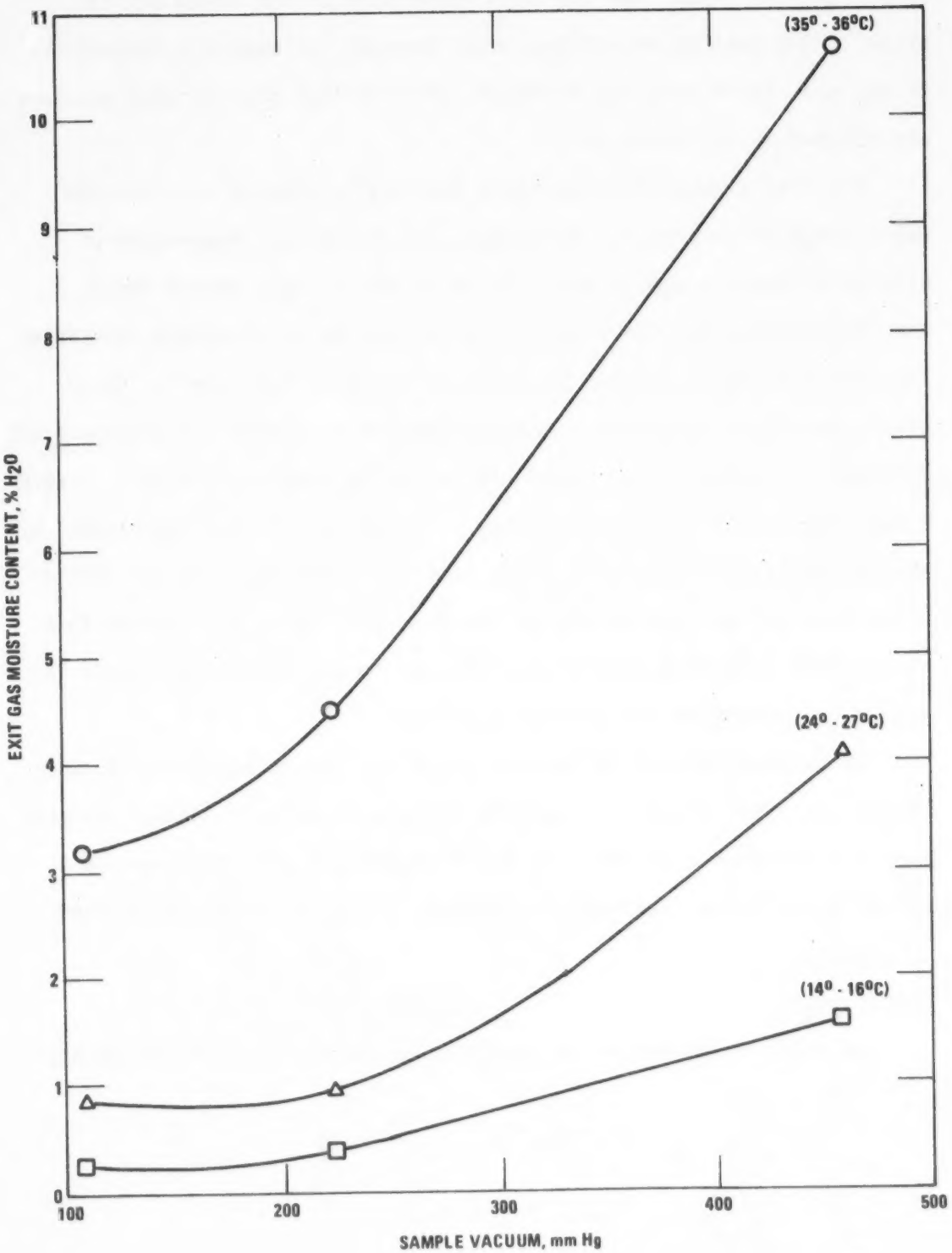

Figure 2. Silica gel exit moisture content versus sample vacuum and exit temperature.

Note that the exit gas from the silica gel impinger has a temperature several degrees higher than the inlet temperature of the silica gel impinger. This temperature difference was somewhat influenced by temperature of the room, but also by the exothermic reaction that occurred when moisture was adsorbed by the silica gel.

Data from similar EPA experiments reported by Johnson[2] substantiate the findings of this study. For example, at an impinger temperature of 21°C (70°F) and a vacuum of about 254 mm Hg (10 in. Hg), Johnson found that the moisture collection efficiency was from 60 to 70 percent, comparing favorably with the 70 percent predicted by the curve in Figure 3. Other values, difficult to compare because temperature conditions and pressures are different, in general, show trends similar to the results noted here. Table 2 shows the results of Johnson's study. In addition, further experiments by Johnson showed that adding one or two more silica gel impingers did little to decrease the moisture content of the final exit gas. This implies that the moisture collection ability of silica gel in the EPA Method 5 train is limited by temperature and pressure conditions.

The moisture content in the exit gas of the silica gel also affects the dry gas meter volume. A 5 percent increase in moisture content produces a similar increase in volume. In source sampling results, this means a 5 percent error in the isokinetic calculations and an error in the emission calculations.

Conclusions

The moisture content of the gas entering the meter box can be greatly

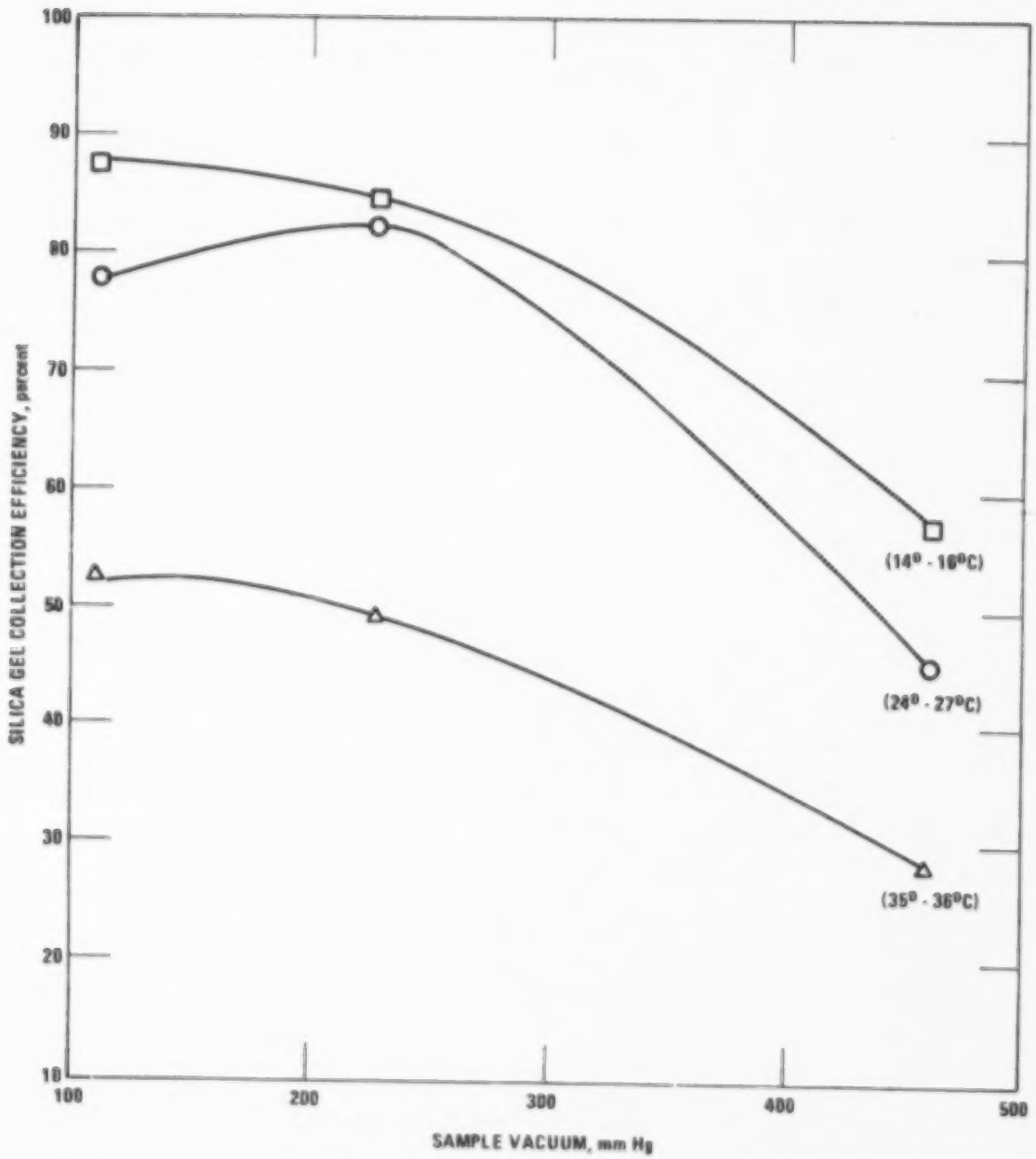

Figure 3. Silica gel moisture collection efficiency versus sample vacuum and exit temperature.

Table 2. Results of W. L. Johnson's Study of Moisture Collection Efficiency of Silica Gel[2]

Run	Vacuum mm Hg	Impinger temp., °C	Exit gas temp., °C	Collection efficiency, %
A	508	-	27.8	38
B	508	18.3	21.1	52
C	508	16.7	20.0	61
D	381	21.7	21.7	46
E	254	21.7	20.6	60
F	152.4	21.7	22.8	52
G	127	16.7	20.0	84

affected by the temperature and vacuum of the sampling train during Method 5 testing. The moisture in the sample gas is incorrectly measured as "dry" gas by the volume meter, and this value is carried through the isokinetic calculations as well as the concentration calculations. A volume measurement error due to moisture in the sample gas directly affects the isokinetic calculations; a 3 percent increase in moisture content of the sample gas produces a similar error in the isokinetic results.

Method 5, as written, stipulates that the sample temperature as it exits the silica gel impinger exit gas be held below 21°C (70°F) and that the sample train vacuum be held under 381 mm Hg (15 in. Hg). These tests show that at these limits the "dry" gas volume error would be less than 2 percent, and a similar error would appear in the isokinetic determination.

It is noted in the text that the temperature of the wet gas in the third dry impinger was 4° to 7°C (7° to 13°F) less than the temperature of the exit gas from the silica gel. This difference is influenced by the heat of adsorption of the silica gel and ambient conditions. These tests were run under steady-state ambient temperature conditions and therefore do not reflect results that may be obtained under field conditions. A better field indicator of acceptable temperature limits for the sample gas would be the dry-bulb temperature in the dry impinger preceding the silica gel. A limit of 15.6°C (60°F) in the impinger would meet the intentions of the present EPA Method 5 specifications.

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1. Title 40 -- Protection of the Environment, Part 60 -- Standards of Performance for New Stationary Sources. Federal Register, 36 (247): 24888, December 23, 1971.

2. Johnson, William L., "Moisture Collection Efficiency of Silica Gel in Stack Sampling Trains," Environmental Protection Agency, National Environmental Research Center, Research Triangle Park, North Carolina unpublished report, 1974.

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

Research Triangle Park, North Carolina 27711

SUBJECT: Spurious Acid Mist Results Caused by Peroxides in Isopropyl Alcohol Solutions Used in EPA Test Method (M-8)

DATE: January 22, 1976

FROM: Dr. Joseph E. Knoll, QAB/EMSL (MD#77)

TO: Mr. Roger T. Shigerhara, EMB/ESED (MD#19)

An evaluation study of EPA Test Method (M-8) for the Determination of Sulfuric Acid Mist and Sulfur Dioxide Emissions from Stationary Sources has been carried out in the Quality Assurance Branch. One result of this study has been the finding that peroxide impurities in the isopropyl alcohol used for acid mist collection can convert sulfur dioxide to sulfuric acid and result in erroneously high acid mist values. The quantities of sulfur dioxide collected as sulfuric acid were of the order of from ten to twenty five percent of the EPA compliance standard. It was independent of the quantity or concentration of sulfur dioxide that had passed through the system and only dependent on the quantity of peroxide, traces of which may occasionally be found in reagent grade isopropyl alcohol.

The following test is tentatively proposed for detecting peroxides in isopropyl alcohol:

> Shake 10 ml of isopropyl alcohol with 10 ml of freshly prepared 10% potassium iodide solution. Prepare a blank by similarly treating 10 ml of distilled water. After one minute, read the absorbance at 352 nm. If absorbance exceeds 0.1, reject alcohol for use.

Peroxides may be removed from isopropyl alcohol by redistilling or by passage through a column of activated alumina. However, it is possible to obtain reagent grade isopropyl alcohol with suitably low peroxide levels from commercial sources, so that rejection of contaminated lots may be a more efficient procedure.

cc: M. R. Midgett

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

SUBJECT: Determination of Isopropanol Loss During Method 8 Simulation Tests

DATE: JUN 25 1976

FROM: Peter R. Westlin, Test Support Section PRW
Emission Measurement Branch (MD 19)

TO: Roger T. Shigehara, Chief, Test Support Section
Emission Measurement Branch (MD 19)

In answer to questions regarding potential loss of isopropanol(IPA) through evaporation and a subsequent error in moisture determination when using Method 8, a laboratory program was undertaken at the IRL during June 17 and 18. A Method 8 sampling train was set up without the glass filter between impinger 1, the IPA impinger, and number 2, the first hydrogen peroxide, H_2O_2, impinger. The third impinger in the train was also a H_2O_2 impinger, while the fourth was left dry. The fifth and last impinger contained silica gel. A standard Method 5 meter box was used to draw and measure the volume of the sample.

Two test runs were completed. For the first run, 200 milliliters(ml) of IPA was placed in the first impinger, 100 ml of H_2O_2 in each of the next two, the fourth impinger was left dry, and about 300 grams(g) of silica gel were placed in the last impinger. About 1420 liters(l) (50 ft^3) of room air were drawn through the train at a flow rate of about 70 liters per minute(lpm) (0.8 cfm).

The results showed a loss of 40 ml of solution in the first impinger, a gain of 15 ml in the second, a 12 ml gain in the third, a negligible gain in the dry impinger, and 20.5 g gain in the silica gel. The net change across the train was 7.5 ml (assuming the mass gained on the silica gel was water). A specific gravity determination showed that the original IPA solution had been prepared incorrectly and was 67% IPA rather than the specified 80% IPA. The solution remaining in the IPA impinger after the test run was shown to be 52% IPA. The 15% loss corrected for total volume change represented a loss of 51 ml of IPA.

For the second run, 100 ml of IPA solution was placed in the first impinger and the rest of the sampling train was the same as for run 1. The IPA solution was prepared as specified in the Federal Register (December 23, 1971) and a specific gravity check of concentration showed the solution to be 73% IPA.

After some 1500 l (54 ft^3) of sample were drawn through the train, the first IPA impinger showed a 50 ml volume loss, the second measured a 75 ml gain, the third showed an 18 ml gain, the third showed a 2 ml gain, and the silica gel mass total increased 11 g. The net volume change across

the train was a 6 ml increase. The final IPA solution concentration was 27.4% representing a loss of about 59 ml of IPA.

These test results indicate that measurement of volume gain in the impingers of the Method 8 for the purposes of calculating sample moisture content is not impaired by any loss of IPA through evaporation. In neither test run was there a net loss of volume from the sample trains. The net gain was approximately equivalent to 0.6% moisture or about 25% relative humidity.

A notable secondary finding of this short study was the great change in IPA solution concentration during a test run. Approximately 1 ml of IPA was removed from first impinger per 30 l of sample gas for each test run. Initial volume of IPA solution or IPA concentration appear to have little effect on this ratio. Some IPA may have been evaporated and condensed farther down the train. More IPA was probably carried through as a mist and collected later.

This IPA loss may be significant if the concentration of IPA gets too low to effectively inhibit oxidation of SO_2 during Method 8 sampling. Joe Knoll was not aware of this potential problem and could not tell me what a lower effective limit of IPA may be. He agreed that it could be a significant problem not only in the possible interference from SO_2 oxidation, but also by meeting the titration end point analysis.

I suggest further work in this problem area be undertaken. Such a project may be suitable for one of the co-op students in the next several months.

COMPARISON OF EMISSION RESULTS FROM IN-STACK FILTER SAMPLING AND EPA METHOD 5 SAMPLING

Peter R. Westlin and Robert L. Ajax*

Abstract

A series of replicate emission tests using in-stack and out-of-stack sampling trains were conducted at each of four fossil-fuel-fired power generation stations. The sampling train used for measuring in-stack particulate included a probe nozzle and an in-stack glass fiber mat filter, followed by a heated probe extension and an out-of-stack filter. The Environmental Protection Agency Method 5 particulate sampling system was used as the out-of-stack sampling train. The two sampling trains were operated simultaneously at approximately the same point in the stack gas streams with no traversing.

The particulate catch from each sampling train was analyzed for particulate mass, sulfate content, organic content, and acidity. For the in-stack train, the results are reported for both the in-stack catch (the particulate obtained from the nozzle and the in-stack filter), and the total catch (the in-stack particulate, plus the particulate washed from the probe extension and the out-of-stack filter).

The tests at two coal-fired units with electrostatic precipitators and an oil-fired unit with no control device resulted in the out-of-stack train catch exceeding the in-stack catch, in each case. The difference varied with the sulfur content of the fuel and ranged from 10 mg/dscm at the unit firing 0.3% sulfur oil, to 112.6 mg/dscm at the unit firing 3% sulfur coal. The measured sulfate did not, however, fully account for this difference.

Opposite results were obtained at a second oil-fired unit with a wet limestone scrubber. At this unit, which was burning 2.5% sulfur fuel, the in-stack catch was significantly greater than the out-of-stack train catch (421.3 mg/dscm versus 217.6 mg/dscm respectively). This difference was the apparent result of a reaction occurring on the wet in-stack filter.

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Presented at the annual APCA Meeting, June 1975

Introduction

During the summer of 1973 the Emission Measurement Branch of the Environmental Protection Agency (EPA) undertook a project in which particulate emissions were sampled with in-stack filter and out-of-stack filter sampling trains.

The purpose of the project was to obtain and compare particulate emission sampling results using sampling trains in which all components except filter location were identical. The equipment used for measuring the in-stack particulate, as shown in Figure 1, consisted of a probe nozzle and an in-stack filter, followed by a probe extension, and an out-of-stack filter. The EPA Method 5 sampling train,[1] shown in Figure 2, was used to measure particulate out-of-stack at 120°C. The two trains were operated simultaneously side-by-side at approximately the same point in the stack gas streams at each of four fossil-fuel-burning power generating stations. Two plants were coal-fired with electrostatic precipitators and two plants were oil-fired, one using a wet limestone scrubber, and the other having no supplementary emission control. The sulfur content of the fuels ranged from 0.29 to 3.3 percent.

The particulate catch from each sampling system was analyzed for particulate mass, sulfate content, organic content, and acidity. The results are reported for the in-stack catch (the particulate obtained from the nozzle and the in-stack filter), the total in-stack (the in-stack particulate plus the particulate washed from the probe extension downstream and the out-of-stack filter), and the EPA Method 5 catch (the particulate from the nozzle, the probe, and the out-of-stack filter). The impinger catch results are not reported in this paper as the dry or front half results were of concern in this project.

Methods

A special dual-probe sampling box was constructed to house the two sampling trains and to allow for simultaneous operation of both systems. Two equal-length sampling probes were employed side-by-side with the probe tips approximately 10 centimeters apart. No provisions were made for traversing of the stack cross-section as only relative concentrations were desired. Although only one point was sampled, isokinetic conditions were maintained. A pitot tube was attached to the EPA Method 5 sampling probe to permit velocity head measurements, and adjustments in the sampling rate of each train were made every five minutes during sampling to maintain isokinetic sampling conditions. Other measurements recorded at regular intervals included stack temperature, dry gas volume, meter vacuum, gas meter temperatures, orifice pressure drop, and sample box temperature. Sample box temperature in the enclosure housing the box filters was carefully monitored and maintained at or above 120°C. The sample box temperature was measured with a thermocouple located in the downstream half of one of the box filters.

The in-stack sampling train was composed of a button-hook sampling nozzle; a 5.7 cm diameter glass-fiber mat filter and an in-stack filter holder; a heated, glass-lined probe; a second 7.6 cm diameter glass-fiber

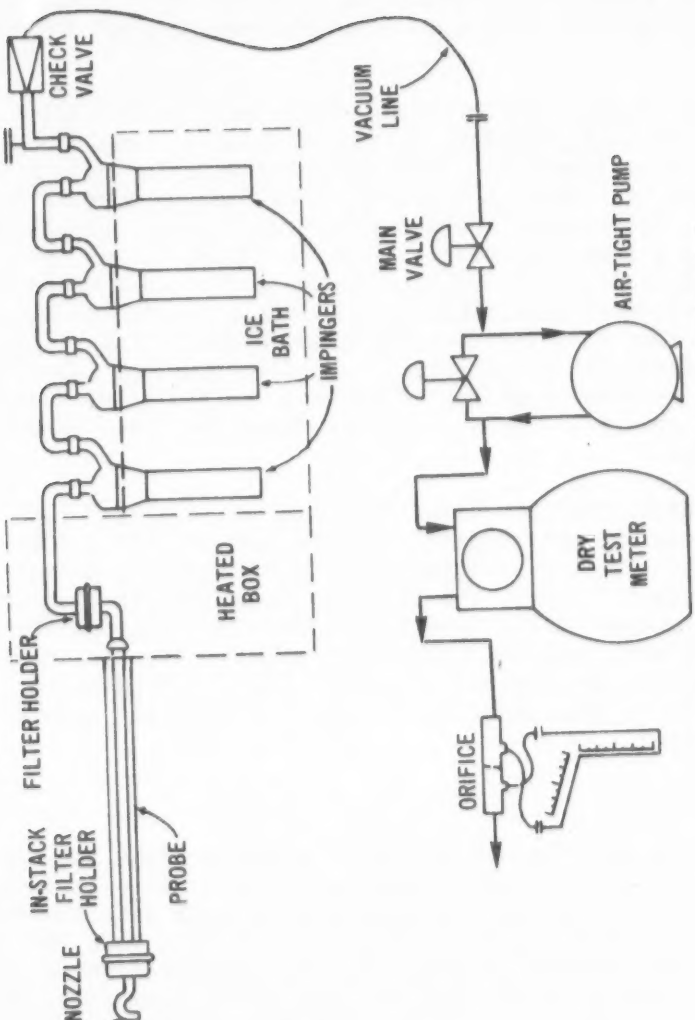

Figure 1. In-stack particulate sampling train.

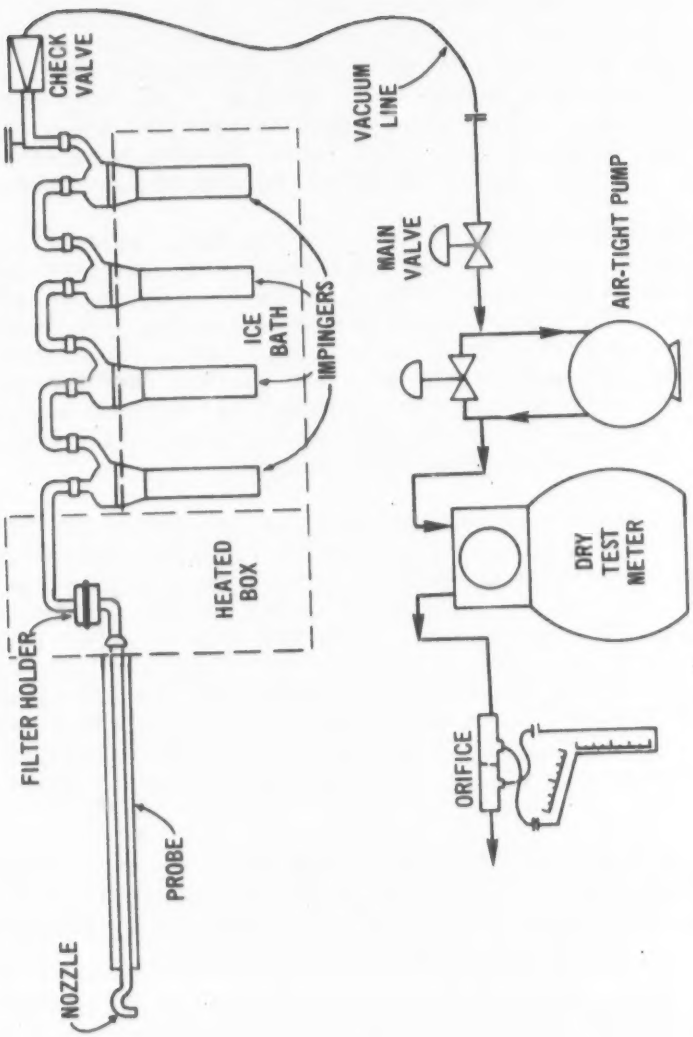

Figure 2. EPA method 5 particulate sampling train.

mat filter in a heated sample box; and condensation impingers. This arrangement along with the pump and metering equipment is displayed in Figure 1. The EPA Method 5 train (Figure 2) was described in the December 23, 1971 Federal Register.[1]

Cleanup procedures were as prescribed for Method 5 in the Federal Register except that the nozzle tip and the filter holder upstream of the in-stack filter were cleaned with acetone and stored separately from the rest of the probe. The probes were rinsed and brushed with acetone, and the catch was saved for analyses. The out-of-stack filters and the in-stack filters were stored in glass petri dishes. The filter holders were rinsed with acetone as were the impingers after the water condensate was saved.

Each sample was carefully analyzed for particulate mass, sulfate content, organic content, and acidity, except for one test where only mass and sulfate were analyzed. The samples were divided into aliquots in order to obtain all the necessary information. The acetone solutions were divided into three aliquots: the first was used to determine mass of particulate, the second was titrated for acidity and for sulfate as SO_4, and the last was extracted for organic materials. The filters were first weighed for particulate mass and then divided in half: one half used to determine organic materials, and the other half analyzed for acidity and sulfate contents.

Particulate mass was determined gravimetrically after proper dessication. Sulfate ($SO_4^=$) content was determined using the thorin titration technique. Ether-chloroform extraction was used to establish the organic content of each sample and an acid-base titration was employed to determine the acid content of each sample. The analytical results were expressed in milligrams (mg) for particulate, $SO_4^=$, and organics; and in milliequivalents (meq) of H_2SO_4 for acidity. These analytical results were then converted into concentration units--milligrams per dry standard cubic meter (mg/dscm)--for statistical analyses and reporting. The sulfate catch was assumed to exist as sulfuric acid, and the concentration of sulfate was expressed as mg/dscm of $H_2SO_4 + 2H_2O$.[2]

The statistical significance of differences between the various data sets was determined by the t-test. For the purposes of this report, a 0.05 percent probability level was set as the minimum of acceptance or rejection of the hypothesis.

Results

Tables 1 through 4 show emission concentrations as determined by the emission tests at the four power plant facilities. Comparisons are made between the EPA Method 5 concentrations and the in-stack concentrations using data obtained during simultaneous, single point sampling. The pollutant emission data and the oxygen data supplied by plant personnel were also used to estimate the emission rate from grams per standard cubic meter to grams per million calories. The oxygen measurements were made at sampling points other than the particulate sampling points and as a result the emission rates are only approximate values.

Facility A

Table 1 shows the data obtained at facility A, a coal burning power plant employing an electrostatic precipitator. The coal being fired had a reported sulfur content of 3.3 percent. Only particulate mass and sulfate concentrations were determined for this facility. The average EPA Method 5 particulate emission concentration was 129.8 gm/dscm corresponding to an approximate emission rate of 0.49 grams per million calories ($g/10^6$ cal).[3] As shown in Table 5, the average particulate concentration determined from the EPA Method 5 train was significantly greater than the particulate catch for the in-stack sampling train, 129.8 versus 17.2 mg/dscm. Adding the probe wash and the filter catch downstream of the in-stack filter to the in-stack catch produced a total particulate concentration of 124.6 mg/dscm which was not significantly different from the Method 5 dry particulate concentrations.

Similarly, the sulfate found in the EPA Method 5 train, 76.3 mg/dscm, was significantly greater than the 4.7 mg/dscm in-stack catch. The total catch of the in-stack train indicated a sulfate concentration of 60.6 mg/dscm which was not significantly different than the 76.3 mg/dscm EPA Method 5 sulfate catch.

Facility B

The emission concentration data obtained at facility B, an oil-fired power plant with no control devices, are shown in Table 2. Sulfur content of the oil was reported to be 0.29 percent. The average EPA Method 5 particulate emission concentration, 38.8 mg/dscm, was significantly greater than the in-stack particulate concentration of 30.1 mg/dscm. Compared with facility A, however, the actual magnitude of this difference is small: 8.7 vs 112.4 mg/dscm. No significant difference was found between the particulate concentration found in the total in-stack dry train, 42.0 mg/dscm, and that found in the EPA Method 5 equipment. The EPA Method 5 particulate concentration corresponded to an approximate emission rate of 0.05 $g/10^6$ cal for facility B.

The difference between EPA Method 5 sulfate concentration, 13.6 mg/dscm, and the in-stack sulfate concentration, 8.4 mg/dscm, was small and was not statistically significant. Similarly, the total in-stack train sulfate concentration of 15.7 mg/dscm was not significantly different from the EPA Method 5 catch, 13.6 mg/dscm.

The organic matter concentration of the EPA Method 5 catch was 12.8 mg/dscm, a level significantly greater than the 7.9 mg/dscm found in the in-stack filter assembly, but significantly less than the 17.6 mg/dscm captured in the total in-stack dry sampling train. The acidity concentration of the EPA Method 5 sampling train, 18.9 mg/dscm of H_2SO_4, was significantly greater than both the in-stack concentration, 3.4 mg/dscm, and the total in-stack sampling train concentration, 10.7 mg/dscm.

Facility C

Facility C was a coal-fired boiler using coal reported at 0.85 percent sulfur and controlling emissions with an electrostatic precipitator. Neither the difference between the dry particulate concentration of the EPA Method 5 train, 226.2 mg/dscm, and the particulate concentration of the in-stack train, 207.9 mg/dscm, nor the difference between the EPA Method 5 particulate concentration and the total in-stack train concentration of 223.4 mg/dscm was statistically significant. It is, however, noteworthy that the difference of 18.1 mg/dscm between the EPA Method 5 train catch and the in-stack catch, and the corresponding values of 112.6 and 8.7 mg/dscm found at facilities A and B respectively, each show a consistent relationship to the fuel sulfur content--0.85, 3.3, and 0.3% sulfur for facilities C, A, and B respectively. This relationship is shown graphically in Figure 3. This is in spite of the fact that the average EPA Method 5 particulate concentration at facility C is equivalent to an approximate emission rate of 0.34 g/10^6 cal which differs from facility B by a factor of 7.

Sulfate emissions for facility C found using the EPA Method 5 train averaged 5.7 mg/dscm, a level significantly greater than the concentration determined from the catch of the in-stack sampling train, 2.9 mg/dscm. The sulfate concentration of the EPA Method 5 catch was not significantly different from the sulfate concentration, 4.7 mg/dscm, of the total in-stack dry sampling train catch. The average EPA Method 5 organic concentration for facility C, 12.4 mg/dscm, was not significantly different from the in-stack organic concentration of 13.5 mg/dscm. Neither was it different from the total in-stack train concentration of 16.4 mg/dscm. The acidity concentration (H_2SO_4) of the EPA Method 5 train was small, 3.2 mg/dscm, for facility C but was significantly greater than the acidity concentration found by the in-stack sampling train, 2.0 mg/dscm. When the back catch was added to the in-stack concentration, the resulting total in-stack acidity concentration was 4.0 mg/dscm. This number was significantly greater than the acidity concentration obtained by the EPA Method 5 sampling assembly. Note that the actual magnitudes of the components--sulfates, organics, and acidity--are relatively small and are less than about 5 percent of the total particulate mass for both the EPA Method 5 catch and the in-stack catch.

Facility D

Facility D was an oil-fired steam generating station using oil with a sulfur content of 2.45 percent. The plant employed a limestone scrubber as the emission control system. No reheat device was present in the gas stream prior to the sampling location. This, along with apparent problems in the demister resulted in an exhaust gas stream which was supersaturated with moisture. The mist caused some problems in sampling and may be the source of the anomalies in the comparison results that follow. The particulate concentration found by the EPA Method 5 system was 217.6 mg/dscm, significantly less than the particulate catch of the in-stack filter, 421.3 mg/dscm. The EPA Method 5 particulate concentration corresponded to an approximate mass emission rate of 0.31 g/10^6 cal for facility D. The total in-stack train particulate concentration was 727.2 mg/dscm.

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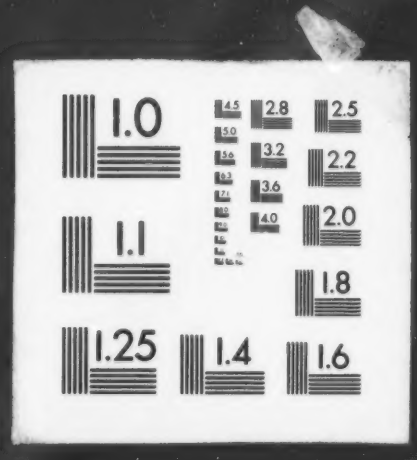

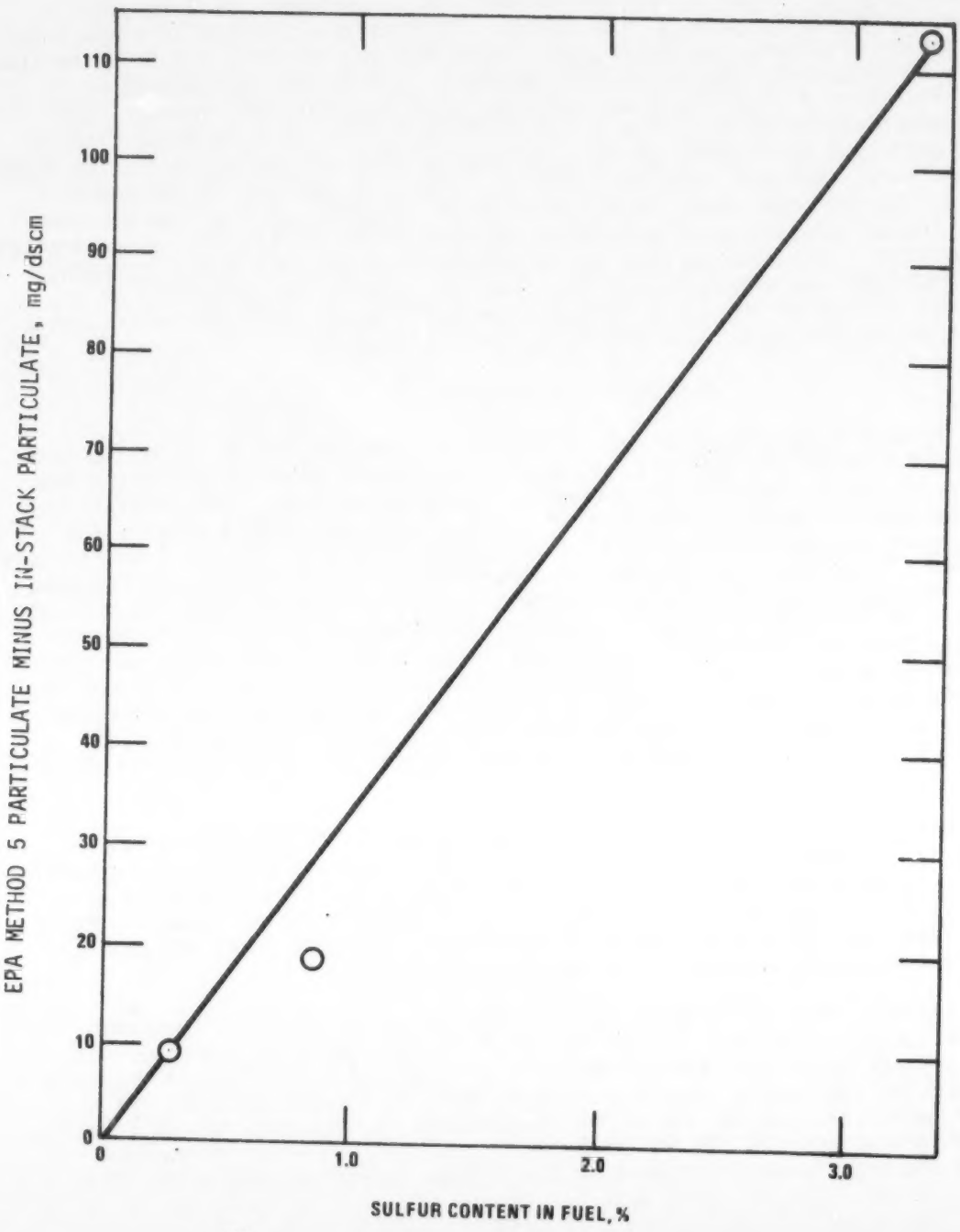

Figure 3. EPA method 5 particulate minus in-stack particulate versus sulfur content in fuel.

The results of the comparison of sulfate concentrations showed different relationships than for the particulate concentrations. The sulfate concentration ($H_2SO_4 + 2H_2O$) in the EPA Method 5 train averaged 119.2 mg/dscm, a value significantly greater than the 18.4 mg/dscm found in the in-stack filter train. Further, the EPA Method 5 value was greater than the total in-stack train sulfate concentration of 55.5 mg/dscm. The acidity concentration of the EPA Method 5 train, 36.6 mg/dscm, was also significantly greater than the concentration found in the in-stack filter train, 10.2 mg/dscm, although no significant difference was observed between the acidity concentration found in the total in-stack sampling train, 31.7 mg/dscm, and the EPA Method 5 train. On the other hand, comparisons of the organic catch of the two sampling trains resulted in relationships similar to the particulate concentration comparisons. The average organic concentration of the EPA Method 5 train, 60.7 mg/dscm, was significantly less than the organic concentration of the in-stack filter train, 120.9 mg/dscm, for facility D. The same relationship was true for the total in-stack train concentration of 137.9 mg/dscm.

Evaluation of Results

Various combinations of the different portions of the total particulate catch of each of the sampling trains were studied in order to determine the source of the differences between measured concentrations. One combination studied was designed to determine if sulfates as $H_2SO_4 + 2H_2O$ make up the difference between in-stack particulate catch and the EPA Method 5 particulate catch.[3] To do this, the EPA Method 5 particulate concentration was compared with the sum of the EPA Method 5 sulfate catch plus the in-stack non-sulfate catch. If these new concentrations were found not to be significantly different, then the difference between the in-stack particulate catch and the EPA Method 5 particulate catch could be attributed to the sulfate caught in the EPA Method 5 sampling train. A similar analysis was done to determine if the difference between the in-stack sampling train catch and the EPA Method 5 catch could be condensible organic matter for those tests in which organic data were available.

For facility A, the coal-burning power plant with 3.3 percent sulfur coal, the comparison of the sulfate test showed that a significantly greater amount of material was caught in the EPA Method 5 train than could be accounted for by the sulfate as $H_2SO_4 + 2H_2O$ in the EPA Method 5 catch. In this case, the difference between in-stack filterable material and EPA Method 5 catch was apparently not all sulfate matter.

The data from facility B, an oil-fired generator with 0.29 percent sulfur fuel, showed that no significant difference could be found between the in-stack catch plus the EPA Method 5 sulfate catch and the EPA Method 5 dry particulate catch. Thus the sulfate found in the EPA Method 5 train could have accounted for the difference between the in-stack dry particulate concentration and the EPA Method 5 concentration. A similar comparison using the organic catch instead of the sulfate showed, however, that the

difference between the in-stack catch and the EPA Method 5 catch could also have been the organic matter found in the EPA Method 5 dry particulate catch which could also have accounted for the difference. This indicates that the variation in the data had as great an influence on the statistical comparison results as did the sulfate or organic catch of the EPA Method 5 sampling train.

The coal-burning power station, facility C, firing 0.85 percent sulfur fuel produced a comparison of emissions results similar to that of facility B. That is, either the sulfate or the organic matter found in the EPA Method 5 particulate catch could have accounted for the differences between the EPA Method 5 particulate concentrations and the in-stack particulate concentrations. This is as expected since there was no significant difference between in-stack particulate collections and EPA Method 5 collection for this site.

Test results of the emissions from site D do not fall into the pattern set previously by the other three test sites. Comparisons of concentrations using the sulfate data or the organic data produced no significant results, as might be expected. The particulate concentrations from the in-stack filter were significantly greater than the dry particulate concentration from the EPA Method 5 train and could not be accounted for with either the sulfate catch or the organic catch.

Conclusions

The in-stack sampling train does not produce results equivalent to the EPA Method 5 sampling train results at all power plant sites. At two power plants where samples were collected in dry stack gases, the in-stack filter tended to collect less material than the EPA Method 5 sampling train. There was no significant difference between the particulate catch of the two trains at a third power plant with dry stack gas and low sulfur fuel. At another site where stack gases were supersaturated with water following a wet scrubber, the in-stack filter collected considerably more particulate than the EPA Method 5 train.

The magnitude of the differences in the material collected by the in-stack filter and the EPA Method 5 train was much greater for the high sulfur fuel power stations than for the low sulfur fuel power plants and showed a consistent relationship to the fuel sulfur content. The differences in the amounts were, however, neither directly attributable to the sulfates found in the EPA Method 5 catch nor to organic matter. Particulate matter collected outside the stack, downstream of the in-stack filter made up the difference between the in-stack catch and the EPA Method 5 catch, but no definite conclusion as to what this material was and why it passed the in-stack filter, can be drawn from this study.

As for the cause of the high in-stack filter catch compared to the EPA Method 5 catch in wet stack gases, chemical reaction between the mineral scrubbing medium and the sulfur oxides in the gas stream may be occurring. These reactions may occur in the stack gas streams, or the sulfur oxides may react with the minerals and the moisture on the wet filter surface of the

in-stack filter. These salts would not be collected on the EPA Method 5 filter as this filter is heated above the dew-point of water and is relatively dry.

Further study in the area of in-stack filters in wet gas streams should answer these questions. Other types of sampling methods may be found more appropriate under these conditions.

References

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TABLE 1

SUMMARY OF EMISSION CONCENTRATIONS FOUND DURING SIMULTANEOUS IN-STACK AND EPA METHOD 5 EMISSION TESTS AT A COAL-FIRED POWER PLANT[a] (mg/dscm)

	Particulate concentration			$H_2SO_4 + 2H_2O$ concentration		
Run	EPA[b]	In-stack	Total[c]	EPA[b] Dry	In-stack	Total[c]
1	93.9	10.1	97.5	43.8	1.7	5.0
2	155.0	20.5	190.8	92.7	2.2	26.2
3	156.2	30.6	133.1	80.9	3.6	108.0
4	65.7	22.7	127.5	33.0	14.3	87.2
5	175.3	8.4	76.3	114.6	2.5	52.7
6	133.7	10.7	122.2	93.0	3.9	84.3
Average	129.8	17.2	124.6	76.3	4.7	60.6

[a]Sulfur content of coal = 3.3 percent, average stack temperature = 139°C.

[b]Based on catch of EPA Method 5 sampling train.

[c]Based on sum of catches of in-stack filter and probe and dry filter of in-stack filter.

TABLE 2

SUMMARY OF EMISSION CONCENTRATIONS FOUND DURING SIMULTANEOUS IN-STACK AND EPA METHOD 5 EMISSION TESTS AT AN OIL-FIRED POWER PLANT[a]
(mg/dscm)

Run	Particulate Concentration			$H_2SO_4 + 2H_2O$ Concentration			Organic Concentration			Acidity(H_2SO_4)Concentration		
	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]
1	44.8	26.0	44.7	30.9	0.6	12.6	19.5	4.4	17.7	28.2	4.3	17.5
2	51.0	24.7	35.8	33.8	0.7	7.9	7.4	6.4	17.0	35.4	3.8	11.9
3	43.2	27.2	50.9	4.5	0.9	14.8	20.7	6.4	23.8	27.9	3.2	17.0
4	42.5	25.5	42.6	3.9	0.6	9.5	19.1	9.5	26.5	30.2	4.1	11.5
5	43.6	28.2	47.4	5.8	0.7	13.6	16.2	9.5	24.3	24.9	9.2	20.1
6	26.8	28.6	34.1	13.2	13.9	24.0	10.1	6.5	10.3	12.7	3.6	18.4
7	25.5	22.5	37.1	4.5	13.3	22.1	5.6	7.3	18.5	19.4	3.8	9.8
8	35.1	23.7		3.4	13.5	20.1	15.0	4.7		34.8	3.3	8.8
9	49.9	50.9	55.0	20.1	22.6	24.4	17.3	10.3	14.2	3.4	1.2	2.9
10	48.6	45.7		18.3	21.2	23.4	12.1	14.7		2.3	1.0	2.0
11	28.1	27.8	34.2	12.0	11.9	13.2	5.0	4.2	7.4	3.0	1.5	5.4
12	27.1	29.9	37.8	12.5	0.7	2.8	6.1	10.9	16.9	4.3	2.0	3.1
Average	38.8	30.1	42.0	13.6	8.4	15.7	12.8	7.9	17.6	18.9	3.4	10.7

a Sulfur content of oil = 0.29 percent, average stack temperature = 168°C.

b Based on catch of EPA Method 5 sampling train.

c Based on sum of catches of in-stack filter and probe and dry filter downstream of the in-stack filter.

TABLE 3

SUMMARY OF EMISSION CONCENTRATIONS FOUND DURING SIMULTANEOUS IN-STACK AND EPA METHOD 5 EMISSION TESTS AT A COAL-FIRED POWER PLANT[a]
(mg/dscm)

Run	Particulate Concentration			$H_2SO_4 + 2H_2O$ Concentration			Organic Concentration			Acidity(H_2SO_4)Concentration		
	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]
1	205.1	161.0	167.6	3.3	2.6	4.8	12.7	32.0	34.4	2.5	1.8	3.6
2	159.3	170.5	180.8	3.9	1.8	2.1	12.7	12.4	16.6	1.8	1.7	3.4
3	183.0	53.6	170.3	5.7	6.8	9.3	9.5	7.8	18.8	1.8	1.8	3.7
4	265.6	227.4	233.8	6.9	2.6	4.4	17.1	8.0	10.5	2.9	2.0	3.9
5	147.4	131.2	136.4	5.1	1.9	2.2	13.6	8.3	10.4	3.3	2.9	5.9
6	310.4	316.9	320.4	5.5	2.8	4.4	12.5	10.8	12.3	2.5	1.4	2.9
7	265.6	262.9	267.0	8.0	3.1	5.1	17.0	13.2	15.1	2.4	2.4	4.0
8	279.5	321.8	326.3	5.7	1.5	3.3	10.7	23.4	25.0	3.9	2.4	4.1
9	216.9	201.1	206.0	4.6	3.1	5.2	16.1	17.6	19.7	3.3	2.4	4.0
10	204.6	213.2	218.6	5.7	4.8	6.6	7.4	15.2	17.6	4.7	2.4	4.0
11	275.6	235.5	245.1	9.9	3.0	5.9	14.3	12.5	13.8	6.6	1.8	4.6
12	220.1	222.8	235.8	5.7	1.9	4.1	7.2	11.0	13.2	3.5	1.8	3.5
13	207.2	184.6	195.8	4.2	1.8	3.6	10.7	2.9	4.5	2.6	1.8	3.7
Average	226.2	207.9	223.4	5.7	2.9	4.7	12.4	13.5	16.4	3.2	2.0	4.0

a Sulfur content of coal = 0.85 percent, average stack temperature = 199°C.

b Based on catch of EPA Method 5 sampling train.

c Based on sum of catches of in-stack filter and probe and dry filter downstream of the in-stack filter.

TABLE 4

SUMMARY OF EMISSION CONCENTRATIONS FOUND DURING SIMULTANEOUS IN-STACK AND EPA METHOD 5 EMISSION TESTS AT AN OIL-FIRED POWER PLANT[a]
(mg/dscm)

	Particulate Concentration			$H_2SO_4 + 2H_2O$ Concentration			Organic Concentration			Acidity(H_2SO_4)Concentration		
Run	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	Total[c]	EPA[b] dry	In-stack	total[c]	EPA[b] dry	In-stack	Total[c]
1	245.3	52.4	167.8	102.6	7.3	166.0	62.2	6.4	74.9	30.3	11.1	55.3
2	101.9	314.4	825.6	88.0	9.0	25.4	37.4	74.5	88.9	27.5	7.8	30.2
3	170.9	425.7	868.0	173.4	21.5	36.2	58.8	102.6	111.0	46.1	11.2	30.2
4	68.6	390.7	603.1	53.8	8.0	179.0	34.3	124.8	133.2	21.8	8.1	57.1
5	203.3	482.5	921.0	178.8	27.2	30.5	70.4	141.6	153.9	43.6	14.3	33.5
6	180.6	483.8	618.7	78.5	20.3	24.9	40.0	132.1	139.6	27.4	11.6	21.1
7	419.8	476.7	905.7	110.8	16.5	19.2	78.5	209.1	233.7	53.3	12.3	30.7
8	171.4	525.6	713.5	112.6	18.0	21.8	63.7	143.5	158.1	42.1	8.7	19.2
9	211.3	425.7	788.8	107.8	19.3	40.4	79.9	117.6	128.2	47.3	9.6	32.7
10	432.0	632.8	1042.8	127.5	33.0	54.4	68.7	162.5	171.2	17.1	14.5	34.7
11	279.8	644.9	930.8	159.5	30.0	48.7	65.2	142.6	156.9	41.9	5.5	20.8
12	126.9	200.7	340.7	136.8	11.3	19.3	69.1	93.7	101.7	40.3	8.2	12.4
Average	217.6	421.3	727.2	119.2	18.4	55.5	60.7	120.9	137.6	36.6	10.2	31.7

[a] Sulfur content of oil = 2.45 percent, average stack temperature = 60°C.

[b] Based on catch of EPA Method 5 sampling train.

[c] Based on sum of catches of in-stack filter and probe and dry filter downstream of in-stack filter.

TABLE 5

RESULTS OF t-TEST COMPARISONS OF CONCENTRATION DETERMINED FROM IN-STACK AND EPA METHOD 5 SAMPLING AT FOSSIL FUEL POWER PLANTS

Facility	Particulate Mass	Sulfate ($H_2SO_4 + 2H_2O$)	Organic	Acidity(H_2SO_4)
A	Method 5[a] > In-stack[b] Method 5 = Total[c]	Method 5 > In-stack Method 5 = Total		
B	Method 5 > In-stack Method 5 = Total	Method 5 = In-stack Method 5 = Total	Method 5 > In-stack Method 5 < Total	Method 5 > In-stack Method 5 > Total
C	Method 5 = In-stack Method 5 = Total	Method 5 > In-stack Method 5 = Total	Method 5 = In-stack Method 5 = Total	Method 5 > In-stack Method 5 < Total
D	Method 5 < In-stack Method 5 < Total	Method 5 > In-stack Method 5 > Total	Method 5 < In-stack Method 5 < Total	Method 5 > In-stack Method 5 = Total

a Based on particulate catch of EPA Method 5 dry sampling train.

b Based on particulate catch of in-stack filter.

c Based on sum of particulate catch of in-stack filter and probe and filter downstream of in-stack filter.

EPA METHOD 5 SAMPLE TRAIN CLEAN-UP PROCEDURES

Clyde E. Riley*

Introduction

In the performance of particulate source emission tests, an important procedure affecting the accuracy is sample recovery. Accurate results are not possible unless proper procedures are conscientiously applied in recovering and quantitatively transferring particulate matter from the sample train to the storage container. Often, however, these procedures receive only minimum attention. Well-trained and highly experienced technical staff are normally employed to design and oversee the performance of a test and the writing of a test report while, in contrast, the least experienced personnel are often assigned sole responsibility with limited guidance for the recovery of sample from the train--a task which includes a high potential for producing significant errors.

The accuracy of sample recovery procedures are, of course, not only dependent on the physical transfer of sample from the train to the storage containers; the procedures also involve the selection of proper equipment, use of proper materials, application of proper cleaning, handling, and shipping techniques, and an overall awareness of the importance of each phase of the sample handling procedure. The following guidelines describe procedures which are employed by the Emission Measurement Branch to assist in minimizing sources of error in EPA Method 5 sample train cleanup. These are presented here, both to call attention to the degree of detail which must be considered in sample recovery, and to make the procedures available to others engaged in source sampling. These guidelines do not include techniques

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Published in Stack Sampling News 3(1): 4-7, July 1975

for analysis of the impinger catch or any specific procedures other than those necessary for analyzing the sample for mass only. Also, it should be noted that these procedures are not regulatory requirements; rather, they are procedures to be used by contractors employed by the Emission Measurement Branch. Although these procedures reflect the collective experience gained by EMB in the conduct of several hundred source tests, we recognize that other source sampling groups may use different cleanup techniques. It is hoped, therefore, that this publication will provide the impetus to others to publish such alternate or improved techniques.

Pretest Preparation

1. Brushes and sample recovery support items shall be properly cleaned and enclosed in dust-free packaging before being used in the sample recovery operations. This includes the sample containers as well as the sample collector glassware.

2. Sample containers to be used for the liquid samples shall be Type I, chemically-resistant, borosilicate narrow-mouth glass bottles (500 mls. or 1000 mls. size). Screw-cap closures with Teflon[1] rubber-backed liners shall be used on all such sample containers. Use of any other type liquid sample container, closure, or liner shall be verified acceptable prior to use.

3. Glass or plastic petri dishes shall be used to contain the filter samples, unless otherwise specified by EPA.

4. Pre-weighed indicating silica gel shall be acceptable only if the containers are completely full and tightly sealed.

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[1]Trade Name

5. Only fresh ACS[2] reagent grade chemicals shall be used for sample cleanup and recovery.

6. All reagents and samples shall be stored in sealed, non-contaminating containers. This includes acetone which shall be purchased and stored in glass containers. Only acetone with blank values less than 0.001 shall be acceptable for sample recovery operations.

7. If water is required for cleanup of the probe and filter assembly, it shall be distilled and stored in non-contaminating containers.

Sample Recovery

1. Proper sample recovery procedure begins as soon as the probe is removed from the stack at the completion of the sampling period. When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over the nozzle tip. Do not cap off the probe tip tightly while the sampling train is cooling as this will create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

2. Before moving the sample train to the cleanup site, remove the probe from the sample train, inspect for condensed water, wipe off the silicone grease, if used, and cap the open end of the probe. Be careful not to lose any condensate. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it loosely. Remove the umbilical cord from the last impinger and cap the impinger opening. If a flexible line is used between either the first impinger or condenser and the filter holder, disconnect the line at the filter holder and drain any condensed liquid into the

[2]American Chemical Society

impingers or condenser and remove the line from the impinger. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet and the flexible line, if used. Either ground glass stoppers or their EPA approved equivalent may be used to close these openings.

3. Transfer the probe and filter-impinger assembly to the cleanup area. Exercise care in moving the collection train from the test site to the sample cleanup area to avoid the loss of collected sample or the gain of extraneous particulate matter. This area shall be clean and protected from the wind to minimize the chances of contaminating or losing portions of the sample.

4. Prior to sample cleanup and during disassembly, an inspection shall be made of the individual components of the sample collector. This inspection should reveal whether or not the sample collector was functioning properly. Also by observing the quantity of sample, it can be estimated if a sufficient amount of matter has been collected for proper analysis. Record any items that could possibly affect the results (e.g., cracked or broken glassware, water in the filter holder, unexpected residue, spent silica gel). State whether or not the sample is still valid and give basis.

5. A consistent procedure shall be used for the sample collector disassembly and cleanup. The following order is recommended:

General

a. The sample containers shall be tightly capped after the sample recovery operation. The closure caps shall be sealed to the narrow-mouth containers with shrink bands, plastic tape, or their equivalent.

The glass petri dishes shall be sealed around their circumference with large rubber bands and secured with plastic tape or its equivalent.

b. All samples including blanks shall be assigned individual identification numbers by using pre-numbered EPA sample identification labels. Where more than one container is needed to contain a given sample, each additional container shall be assigned the same basic identification number. All such multiple containers shall be further marked to indicate the total number of containers used for that sample and which container of the series each represents (examples 1 of 3, 2 of 3, etc.).

c. After the recovery operation, the volume of all liquid samples including rinses shall be documented either by using graduated sample bottles and recording the sample volume on the recovery sheet or by permanently marking the sample container and/or label to indicate the liquid level. By doing this, the laboratory will be able to determine whether or not sample leakage occurred during transport.

d. A 200 ml blank reagent sample shall be collected for each lot of rinse reagents used. Representative blank samples of the acetone or other solvents, distilled water (if used), and preweighed filters (quantity three) shall be collected during the test program. The acetone and water samples shall be analyzed to determine the amount of contamination attributed to the sample reagents.

Filter - Remove the filter holder and inspect the filter mat for punctures or tears before removing and placing it in an identified glass or plastic petri dish container. Use a pair of parallel tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Quantitatively remove any particulate matter and/or filter media which may adhere to the filter holder or support by carefully using a dry nylon bristle brush, rubber policeman, or a sharp-edged blade. Place this matter into the same container as the filter. Seal the container as described in the General Section.

Probe - It is recommended that two people be used to clean the probe to minimize altering the sample. The probe cleanup and disassembly shall be conducted in the following order. Making sure that dust on the outside of the probe or other exterior surfaces does not enter into the sample, quantitatively transfer the particulate matter and condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder to container No. 2. Rinse these components with acetone, distilled water (if required), or other appropriate rinsing solvents that have been approved

by EPA. In all cases, collect a representative blank of the rinse solvents. Specific steps are as follows:

a. Carefully remove the probe nozzle and clean the inside surface by triple rinsing with acetone from a glass wash bottle and brushing twice with a precleaned nylon brush. Continue brushing until the acetone rinse shows no visible particles, after which perform a final rinse of the inside surface with acetone.

b. Brush and rinse with acetone the inside parts of the probe fitting in a similar way, i.e., until the rinse shows no visible particles remaining.

c. Rinse the probe liner with acetone by tilting and squirting acetone into its upper end, while rotating the liner in a 360° manner so that all inside surfaces will be rinsed. Let the acetone drain from the lower end into the sample container. A second acetone rinse shall be performed with the aid of a probe brush. Position the liner as before and squirt acetone into the upper end while pushing the brush through the entire length of the liner using a twisting action. Repeat the brushing and rinsing operation (minimum two times) until no particulate matter remains in the probe liner upon visual inspection. With stainless steel or other metal liners, brush and rinse in the above prescribed manner at least six times; metal liners have small crevices in which particulate matter can be entrapped. Upon completion of the brushing and rinsing operation, rinse the brush with acetone and perform a final acetone rinse on the liner. Collect these rinsings in the same sample container as before.

d. After ensuring that the filter holder has been wipped clean of silicone grease, clean the inside of the front half of the filter holder by double brushing with a nylon bristle brush while rinsing with acetone or brush and rinse until all visible particulate is removed. Make a final rinse of the brush and inside surface of the front half of the filter holder. Again these rinsings are placed in the No. 2 sample container. (Note: Do not rinse or brush the fritted-glass support.)

Silica Gel - Record the color and condition of the indicating silica gel in the last impinger and determine if it is completely saturated. Weigh the used silica gel to the nearest 0.5 gm and determine the amount of moisture collected. The silica gel shall be transferred to a shipping container or discarded if contaminated.

Impinger Catch - If analysis of the impinger catch is not required, discard the liquid after measuring and recording the volume or weight.